RT Window: <None> Peaks/min: <None>
Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Label Methods 1-5: N,R,S,#,U

Comp No.	Compound Name	(Type)	Retention Time Min. (Rel)	R.F.	Conc.
20)	2-Butanone	(T)	11.12(.944)	.13639	10.00

Mass	Abun
43.0	100.00
72.0	Display only
57.0	Display only

Quant Ion: 43.0 Ref Namr: BUTAN2

RT Window: <None> Peaks/min: <None>
Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Label Methods 1-5: N,R,S,#,U

Comp No.	Compound Name	(Type)	Retention Time Min. (Rel)	R.F.	Conc.
21)	Carbon Disulfide	(T)	8.80(.747)	3.82985	10.00

Mass	Abun
75.8	100.00
77.8	9.00

Quant Ion: 75.8 Ref Namr: CARBON

RT Window: <None> Peaks/min: <None>
Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Label Methods 1-5: N,R,S,#,U

Comp No.	Compound Name	(Type)	Retention Time Min. (Rel)	R.F.	Conc.
22)	Iodomethane	(T)	8.78(.745)	8.84562	20.00

Mass	Abun
142.0	100.00
127.0	50.00
141.0	12.00

Quant Ion: 142.0 Ref Namr: IODOME

RT Window: <None> Peaks/min: <None>
Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Label Methods 1-5: N,R,S,#,U

Comp No.	Compound Name	(Type)	Retention Time Min. (Rel)	R.F.	Conc.
23)	Vinyl Acetate	(T)	10.47(.889)	.48229	10.00

Mass	Abun
42.8	100.00
85.8	10.00

Quant Ion: 42.8 Ref Namr: VINYL

RT Window: <None> Peaks/min: <None>
Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Label Methods 1-5: N,R,S,#,U

Comp No.	Compound Name	(Type)	Retention Time Min. (Rel)	R.F.	Conc.
24)	*1,4-Difluorobenzene	(I)	12.74(1.000)	1.00000	10.00

Mass	Abun
114.1	100.00
63.1	24.57
88.1	21.00

Quant Ion: 114.1 Ref Namr: DIFLUO

RT Window: <None> Peaks/min: <None>
Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Label Methods 1-5: N,R,S,#,U
User Label: 1,4-Difluorobenzene

Comp No.	Compound Name	(Type)	Retention Time Min. (Rel)	R.F.	Conc.
25)	Carbon Tetrachloride	(T)	11.88(.932)	.89246	10.00

Mass	Abun
116.9	100.00
118.9	95.00
47.0	31.00

Quant Ion: 116.9 Ref Namr: CCL4

RT Window: <None> Peaks/min: <None>
Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Label Methods 1-5: N,R,S,#,U

Comp No.	Compound Name	(Type)	Retention Time Min. (Rel)	R.F.	Conc.
26)	1,2-Dichloroethane-d4	(S)	12.11(.951)	.22629	10.00

Mass	Abun
64.8	100.00
101.8	23.00
66.8	48.00

Quant Ion: 64.8 Ref Namr: DCED4

RT Window: <None> Peaks/min: <None>
Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Label Methods 1-5: N,R,S,#,U
User Label: 1,2-Dichloroethane-d

Comp No.	Compound Name	(Type)	Retention Time Min. (Rel)	R.F.	Conc.
27)	1,1-Dichloropropene	(T)	11.90(.934)	.51409	10.00

Mass	Abun

Mass	Abun
75.0	100.00
110.0	29.80
77.0	31.00

RT Window: <None> Peaks/min: <None>
Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Label Methods 1-5: N,R,S,#,U

Comp No.	Compound Name	(Type)	Retention Time Min. (Rel)	R.F.	Conc.
28)	Methyl-tert-Butyl Ether	(T)	9.83(.772)	0.00000	10.00

Mass	Abun
73.0	100.00
57.0	26.00
45.0	10.00

Quant Ion: 73.0 Ref Namr: MTBE

RT Window: <None> Peaks/min: <None>
Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Label Methods 1-5: N,R,S,#,U

Comp No.	Compound Name	(Type)	Retention Time Min. (Rel)	R.F.	Conc.
29)	Benzene	(T)	12.17(.955)	.73344	10.00

Mass	Abun
78.0	100.00
77.0	25.00

Quant Ion: 78.0 Ref Namr: BENZ

RT Window: <None> Peaks/min: <None>
Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Label Methods 1-5: N,R,S,#,U

Comp No.	Compound Name	(Type)	Retention Time Min. (Rel)	R.F.	Conc.
30)	1,2-Dichloroethane	(T)	12.21(.958)	.23227	10.00

Mass	Abun
62.0	100.00
64.0	33.00
49.0	39.00

Quant Ion: 62.0 Ref Namr: DCE12

RT Window: <None> Peaks/min: <None>
Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Label Methods 1-5: N,R,S,#,U

Comp No.	Compound Name	(Type)	Retention Time Min. (Rel)	R.F.	Conc.
31)	Toluene-d8	(S)	15.05(1.181)	.96588	10.00

Mass	Abun				
98.0	100.00	Quant Ion:	98.0	Ref Namr:	TOLD8
100.0	64.00				

RT Window: <None> Peaks/min: <None>
Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Label Methods 1-5: N,R,S,#,U
User Label: Toluene-d8

Comp No.	Compound Name	(Type)	Retention Time Min.	(Rel)	R.F.	Conc.
32)	Trichloroethene	(T)	13.09	(1.028)	.48837	10.00

Mass	Abun				
130.0	100.00	Quant Ion:	95.0	Ref Namr:	TCE=
95.0	94.50				
97.0	59.00				

RT Window: <None> Peaks/min: <None>
Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Label Methods 1-5: N,R,S,#,U

Comp No.	Compound Name	(Type)	Retention Time Min.	(Rel)	R.F.	Conc.
33)	2-Chloroethyl vinyl ether	(T)	14.41	(1.131)	.06056	10.00

Mass	Abun				
63.0	100.00	Quant Ion:	63.0	Ref Namr:	CHLORO
43.0	73.00				
106.0	28.00				

RT Window: <None> Peaks/min: <None>
Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Label Methods 1-5: N,R,S,#,U

Comp No.	Compound Name	(Type)	Retention Time Min.	(Rel)	R.F.	Conc.
34)	1,2-Dichloropropane	(T)	13.44	(1.055)	.28915	10.00

Mass	Abun				
63.0	100.00	Quant Ion:	63.0	Ref Namr:	DCP12
62.0	82.00				
76.0	46.00				

RT Window: <None> Peaks/min: <None>
Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Label Methods 1-5: N,R,S,#,U

Comp No.	Compound Name	(Type)	Retention Time Min.	(Rel)	R.F.	Conc.

35) Bromodichloromethane (T) 13.90(1.091) .71356 10.00

Mass	Abun
83.0	100.00
85.0	64.00

Quant Ion: 83.0 Ref Namr: BRCL2M

RT Window: <None> Peaks/min: <None>
Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Label Methods 1-5: N,R,S,#,U

Comp No.	Compound Name	(Type)	Retention Time Min. (Rel)	R.F.	Conc.
36)	Dibromomethane	(T)	13.62(1.069)	.25532	10.00

Mass	Abun
173.9	100.00
93.0	81.00
95.0	66.00

Quant Ion: 93.0 Ref Namr: VOA24

RT Window: <None> Peaks/min: <None>
Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Label Methods 1-5: N,R,S,#,U

Comp No.	Compound Name	(Type)	Retention Time Min. (Rel)	R.F.	Conc.
37)	4-Methyl-2-Pentanone	(T)	14.93(1.172)	.07122	10.00

Mass	Abun
42.8	100.00
57.8	39.00
99.8	20.00

Quant Ion: 42.8 Ref Namr: M4PEN2

RT Window: <None> Peaks/min: <None>
Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Label Methods 1-5: N,R,S,#,U

Comp No.	Compound Name	(Type)	Retention Time Min. (Rel)	R.F.	Conc.
38)	trans-1,3-Dichloropropene	(T)	15.62(1.226)	.27702	10.00

Mass	Abun
74.8	100.00
76.8	31.00
48.8	22.00

Quant Ion: 74.8 Ref Namr: TCLPRP

RT Window: <None> Peaks/min: <None>
Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Label Methods 1-5: N,R,S,#,U

Comp Compound Retention Time

Comp No.	Compound Name	(Type)	Retention Time Min. (Rel)	R.F.	Conc.
39)	Toluene	(T)	15.17(1.191)	1.01335	10.00

Mass	Abun
91.1	100.00
92.1	61.00

Quant Ion: 91.1 Ref Namr: TOLUEN

RT Window: <None> Peaks/min: <None>
Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Label Methods 1-5: N,R,S,#,U

Comp No.	Compound Name	(Type)	Retention Time Min. (Rel)	R.F.	Conc.
40)	1,2-Dibromoethane	(T)	16.79(1.318)	.29742	10.00

Mass	Abun
107.0	100.00
109.0	94.70
188.0	5.00

Quant Ion: 107.0 Ref Namr: DIBROM

RT Window: <None> Peaks/min: <None>
Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Label Methods 1-5: N,R,S,#,U

Comp No.	Compound Name	(Type)	Retention Time Min. (Rel)	R.F.	Conc.
41)	cis-1,3-Dichloropropene	(T)	14.62(1.148)	.34242	10.00

Mass	Abun
74.8	100.00
76.8	31.00
48.8	25.00

Quant Ion: 74.8 Ref Namr: CCLPRP

RT Window: <None> Peaks/min: <None>
Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Label Methods 1-5: N,R,S,#,U

Comp No.	Compound Name	(Type)	Retention Time Min. (Rel)	R.F.	Conc.
42)	1,1,2-Trichloroethane	(T)	15.91(1.249)	.16938	10.00

Mass	Abun
97.0	100.00
83.0	86.00
85.0	56.00

Quant Ion: 97.0 Ref Namr: TCE112

RT Window: <None> Peaks/min: <None>
Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Label Methods 1-5: N,R,S,#,U

Comp No.	Compound Name	(Type)	Retention Time Min.	(Rel)	R.F.	Conc.
43)	*Chlorobenzene-d5	(I)	17.68	(1.000)	1.00000	10.00

Mass	Abun
116.8	100.00
118.8	32.00

Quant Ion: 116.8 Ref Namr: CBZD5

RT Window: <None> Peaks/min: <None>
Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Label Methods 1-5: N,R,S,#,U
User Label: Chlorobenzene-d5

Comp No.	Compound Name	(Type)	Retention Time Min.	(Rel)	R.F.	Conc.
44)	Tetrahydrofuran	(T)	15.03	(.850)	0.00000	10.00

Mass	Abun
42.0	100.00
71.0	Display only
72.0	Display only

Quant Ion: 42.0 Ref Namr: THF

RT Window: <None> Peaks/min: <None>
Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Label Methods 1-5: N,R,S,#,U

Comp No.	Compound Name	(Type)	Retention Time Min.	(Rel)	R.F.	Conc.
45)	Bis(Chloromethyl)Ether	(T)	13.89	(.786)	0.00000	6400.00

Mass	Abun
79.0	100.00
49.0	Display only
81.0	Display only

Quant Ion: 79.0 Ref Namr: <None>

RT Window: <None> Peaks/min: <None>
Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Label Methods 1-5: N,R,S,#,U

Comp No.	Compound Name	(Type)	Retention Time Min.	(Rel)	R.F.	Conc.
46)	2-Hexanone	(T)	16.43	(.929)	.07863	10.00

Mass	Abun
42.8	100.00
57.8	40.00
99.8	15.00

Quant Ion: 42.8 Ref Namr: HEXON2

RT Window: <None> Peaks/min: <None>
Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Comp No.	Compound Name	(Type)	Retention Time Min.	(Rel)	R.F.	Conc.
47)	Ethyl methacrylate	(T)	15.80	(.894)	.19894	20.00

Mass	Abun
69.0	100.00
41.0	Display only
99.0	Display only

Quant Ion: 69.0 Ref Namr: APIX19

RT Window: <None> Peaks/min: <None>
Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Label Methods 1-5: N,R,S,#,U

Comp No.	Compound Name	(Type)	Retention Time Min.	(Rel)	R.F.	Conc.
48)	Tetrachloroethene	(T)	16.10	(.910)	1.05323	10.00

Mass	Abun
165.9	100.00
167.9	47.00
129.0	64.00

Quant Ion: 165.9 Ref Namr: PERC

RT Window: <None> Peaks/min: <None>
Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Comp No.	Compound Name	(Type)	Retention Time Min.	(Rel)	R.F.	Conc.
49)	1,3-Dichloropropane	(T)	16.18	(.915)	.31498	10.00

Mass	Abun
76.0	100.00
78.0	32.00

Quant Ion: 76.0 Ref Namr: VOA28

RT Window: <None> Peaks/min: <None>
Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Comp No.	Compound Name	(Type)	Retention Time Min.	(Rel)	R.F.	Conc.
50)	Dibromochloromethane	(T)	16.59	(.938)	.64263	10.00

Mass	Abun
129.0	100.00
127.0	76.00

Quant Ion: 129.0 Ref Namr: DBCM

RT Window: <None> Peaks/min: <None>
Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Comp Compound Retention Time

Comp No.	Compound Name	(Type)	Retention Time Min.	(Rel)	R.F.	Conc.
51)	Chlorobenzene	(T)	17.74	(1.003)	1.00923	10.00

Mass	Abun
112.1	100.00
114.0	32.00
77.0	62.00

Quant Ion: 112.1 Ref Namr: CLB

RT Window: <None> Peaks/min: <None>
Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Comp No.	Compound Name	(Type)	Retention Time Min.	(Rel)	R.F.	Conc.
52)	1,1,1,2-Tetrachloroethane	(T)	17.92	(1.013)	.61154	10.00

Mass	Abun
131.0	100.00
133.0	96.00
119.0	71.00

Quant Ion: 131.0 Ref Namr: VOA33

RT Window: <None> Peaks/min: <None>
Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Comp No.	Compound Name	(Type)	Retention Time Min.	(Rel)	R.F.	Conc.
53)	Ethylbenzene	(T)	18.00	(1.018)	.49399	10.00

Mass	Abun
91.1	100.00
106.1	29.70

Quant Ion: 106.1 Ref Namr: ETHBEN

RT Window: <None> Peaks/min: <None>
Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Comp No.	Compound Name	(Type)	Retention Time Min.	(Rel)	R.F.	Conc.
54)	p-Xylene	(T)	18.23	(1.031)	2.93726	10.00

Mass	Abun
91.1	100.00
106.1	47.00

Quant Ion: 91.1 Ref Namr: XYLN

RT Window: <None> Peaks/min: <None>
Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Comp No.	Compound Name	(Type)	Retention Time Min.	(Rel)	R.F.	Conc.
55)	m-Xylene	(T)	18.23	(1.031)	2.93726	10.00

Mass Abun

91.2 100.00
106.1 48.00

RT Window: <None> Peaks/min: <None>
Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Comp No.	Compound Name	(Type)	Retention Time Min. (Rel)	R.F.	Conc.
56)	o-Xylene	(T)	19.03(1.076)	1.45111	10.00

Mass	Abun
91.1	100.00
106.1	44.00

Quant Ion: 91.1 Ref Namr: XYLN

RT Window: <None> Peaks/min: <None>
Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Comp No.	Compound Name	(Type)	Retention Time Min. (Rel)	R.F.	Conc.
57)	Styrene	(T)	19.07(1.079)	.82965	10.00

Mass	Abun
104.0	100.00
78.0	43.00

Quant Ion: 104.0 Ref Namr: STYREN

RT Window: <None> Peaks/min: <None>
Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Comp No.	Compound Name	(Type)	Retention Time Min. (Rel)	R.F.	Conc.
58)	Bromoform	(T)	19.41(1.097)	.37937	10.00

Mass	Abun
172.9	100.00
170.9	51.00
174.9	50.00

Quant Ion: 172.9 Ref Namr: BROFOR

RT Window: <None> Peaks/min: <None>
Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Comp No.	Compound Name	(Type)	Retention Time Min. (Rel)	R.F.	Conc.
59)	*1,2-Dichlorobenzene-d4	(I)	23.55(1.000)	1.00000	10.00

Mass	Abun
150.0	100.00
152.0	64.00
115.0	38.00

Quant Ion: 150.0 Ref Namr: DCBD4

Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Label Methods 1-5: N,R,S,#,U
User Label: 1,2-Dichlorobenzene

Comp No.	Compound Name	(Type)	Retention Time Min. (Rel)	R.F.	Conc.
60)	Bromofluorobenzene	(S)	20.14(.855)	.97337	10.00

Mass	Abun
95.0	100.00
174.0	Display only

Quant Ion: 95.0 Ref Namr: <None>

RT Window: <None> Peaks/min: <None>
Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Label Methods 1-5: N,R,S,#,U
User Label: Bromofluorobenzene

Comp No.	Compound Name	(Type)	Retention Time Min. (Rel)	R.F.	Conc.
61)	Isopropylbenzene	(T)	19.85(.843)	2.30424	10.00

Mass	Abun
105.0	100.00
120.1	26.60

Quant Ion: 105.0 Ref Namr: VOA40

RT Window: <None> Peaks/min: <None>
Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Comp No.	Compound Name	(Type)	Retention Time Min. (Rel)	R.F.	Conc.
62)	1,1,2,2-Tetrachloroethane	(T)	20.55(.873)	.31823	10.00

Mass	Abun
83.0	100.00
85.0	64.70
131.0	9.20

Quant Ion: 83.0 Ref Namr: CE112

RT Window: <None> Peaks/min: <None>
Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Comp No.	Compound Name	(Type)	Retention Time Min. (Rel)	R.F.	Conc.
63)	Bromobenzene	(T)	20.42(.867)	.70202	10.00

Mass	Abun
77.0	100.00
156.0	69.50
158.0	67.20

Quant Ion: 156.0 Ref Namr: VOA42

Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Comp No.	Compound Name	(Type)	Retention Time Min. (Rel)	R.F.	Conc.
64)	1,2,3-Trichloropropane	(T)	20.59(.874)	.08397	10.00

Mass	Abun
75.0	100.00
77.0	32.00
110.0	34.00

Quant Ion: 110.0 Ref Namr: VOA43

RT Window: <None> Peaks/min: <None>
Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Comp No.	Compound Name	(Type)	Retention Time Min. (Rel)	R.F.	Conc.
65)	n-Propylbenzene	(T)	20.75(.881)	.54787	10.00

Mass	Abun
91.1	100.00
120.0	14.00

Quant Ion: 120.0 Ref Namr: VOA44

RT Window: <None> Peaks/min: <None>
Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Comp No.	Compound Name	(Type)	Retention Time Min. (Rel)	R.F.	Conc.
66)	2-Chlorotoluene	(T)	20.87(.886)	.53287	10.00

Mass	Abun
91.0	100.00
126.0	35.00

Quant Ion: 126.0 Ref Namr: VOA45

RT Window: <None> Peaks/min: <None>
Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Comp No.	Compound Name	(Type)	Retention Time Min. (Rel)	R.F.	Conc.
67)	trans-1,4-dichloro-2-butene	(T)	20.60(.875)	.31496	20.00

Mass	Abun
75.0	100.00
89.0	Display only
124.0	Display only

Quant Ion: 75.0 Ref Namr: APIX19

RT Window: <None> Peaks/min: <None>
Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Label Methods 1-5: N,R,S,#,U

Comp No.	Compound Name	(Type)	Retention Time Min.	(Rel)	R.F.	Conc.
68)	4-Chlorotoluene	(T)	21.12	(.897)	.56787	10.00

Mass	Abun
91.1	100.00
126.0	32.00

Quant Ion: 126.0 Ref Namr: VOA45

RT Window: <None> Peaks/min: <None>
Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Comp No.	Compound Name	(Type)	Retention Time Min.	(Rel)	R.F.	Conc.
69)	1,3,5-Trimethylbenzene	(T)	21.16	(.899)	1.91364	10.00

Mass	Abun
105.0	100.00
120.0	52.52

Quant Ion: 105.0 Ref Namr: VOA47

RT Window: <None> Peaks/min: <None>
Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Comp No.	Compound Name	(Type)	Retention Time Min.	(Rel)	R.F.	Conc.
70)	tert-Butylbenzene	(T)	21.86	(.929)	1.97576	10.00

Mass	Abun
119.1	100.00
91.1	69.00
134.1	30.00

Quant Ion: 119.1 Ref Namr: VOA48

RT Window: <None> Peaks/min: <None>
Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Comp No.	Compound Name	(Type)	Retention Time Min.	(Rel)	R.F.	Conc.
71)	1,2,4-Trimethylbenzene	(T)	21.98	(.934)	1.80746	10.00

Mass	Abun
105.0	100.00
120.0	48.00

Quant Ion: 105.0 Ref Namr: VOA49

RT Window: <None> Peaks/min: <None>
Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Comp No.	Compound Name	(Type)	Retention Time Min.	(Rel)	R.F.	Conc.

72)	sec-Butylbenzene	(T)	22.37(.950)	2.77682	10.00

Mass	Abun
105.1	100.00
134.1	21.00

Quant Ion: 105.1 Ref Namr: VOA50

RT Window: <None> Peaks/min: <None>
Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Comp No.	Compound Name	(Type)	Retention Time Min. (Rel)	R.F.	Conc.
73)	p-Isopropyltoluene	(T)	22.73(.965)	2.29034	10.00

Mass	Abun
119.1	100.00
134.1	28.00
91.0	23.00

Quant Ion: 119.1 Ref Namr: VOA51

RT Window: <None> Peaks/min: <None>
Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Comp No.	Compound Name	(Type)	Retention Time Min. (Rel)	R.F.	Conc.
74)	1,3-Dichlorobenzene	(T)	22.53(.957)	1.13387	10.00

Mass	Abun
146.0	100.00
148.0	60.00
111.0	38.00

Quant Ion: 146.0 Ref Namr: MDCLBZ

RT Window: <None> Peaks/min: <None>
Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Comp No.	Compound Name	(Type)	Retention Time Min. (Rel)	R.F.	Conc.
75)	1,4-Dichlorobenzene	(T)	22.75(.966)	1.09543	10.00

Mass	Abun
146.0	100.00
148.0	64.00
111.0	38.00

Quant Ion: 146.0 Ref Namr: PDCLBZ

RT Window: <None> Peaks/min: <None>
Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Comp No.	Compound Name	(Type)	Retention Time Min. (Rel)	R.F.	Conc.
76)	1,2-Dichlorobenzene	(T)	23.59(1.002)	.99663	10.00

Mass Abun

146.0 100.00
148.0 62.00
111.0 38.00

RT Window: <None> Peaks/min: <None>
Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Comp No.	Compound Name	(Type)	Retention Time Min. (Rel)	R.F.	Conc.
77)	n-Butylbenzene	(T)	23.67(1.005)	2.30917	10.00

Mass Abun
91.1 100.00
92.1 62.50
134.1 27.60

Quant Ion: 91.1 Ref Namr: VOA56

RT Window: <None> Peaks/min: <None>
Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Comp No.	Compound Name	(Type)	Retention Time Min. (Rel)	R.F.	Conc.
78)	1,2-Dibromo-3-Chloropropane	(T)	25.45(1.081)	.04389	10.00

Mass Abun
157.0 100.00
155.0 78.00
75.0 65.60

Quant Ion: 75.0 Ref Namr: VOA57

RT Window: <None> Peaks/min: <None>
Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Comp No.	Compound Name	(Type)	Retention Time Min. (Rel)	R.F.	Conc.
79)	1,2,4-Trichlorobenzene	(T)	27.38(1.163)	.76701	10.00

Mass Abun
180.0 100.00
182.0 94.50
145.0 27.20

Quant Ion: 180.0 Ref Namr: VOA58

RT Window: <None> Peaks/min: <None>
Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Comp No.	Compound Name	(Type)	Retention Time Min. (Rel)	R.F.	Conc.
80)	Hexachlorobutadiene	(T)	27.85(1.183)	1.44748	10.00

Mass Abun
224.9 100.00

Quant Ion: 224.9 Ref Namr: VOA59

226.9	64.00

RT Window: <None> Peaks/min: <None>
Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Comp No.	Compound Name	(Type)	Retention Time Min. (Rel)	R.F.	Conc.
81)	Naphthalene	(T)	27.93(1.186)	.40273	10.00

Mass	Abun
128.1	100.00
129.1	11.00

Quant Ion: 128.1 Ref Namr: VOA60

RT Window: <None> Peaks/min: <None>
Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Comp No.	Compound Name	(Type)	Retention Time Min. (Rel)	R.F.	Conc.
82)	1,2,3-Trichlorobenzene	(T)	28.52(1.211)	.53017	10.00

Mass	Abun
180.0	100.00
182.0	95.00
145.0	28.90

Quant Ion: 180.0 Ref Namr: VOA61

RT Window: <None> Peaks/min: <None>
Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

* Compound is ISTD

Target and Internal Standards

Pentafluorobenzene
Acetone
Acrolein
Acrylonitrile
Bromochloromethane
Bromomethane
2-Butanone
Carbon disulfide
Chloroethane
Chloroform
Chloromethane
Dichlorodifluoromethane
1,1-Dichloroethane
1,1-Dichloroethene
cis-1,2-Dichloroethene
trans-1,2-Dichloroethene
2,2-Dichloropropane
Iodomethane
Methylene chloride
1,1,1-Trichloroethane
Trichlorofluoromethane
Vinyl acetate
Vinyl Chloride

Chlorobenzene-d_5
Bromoform
Chlorodibromomethane
Chlorobenzene
1,3-Dichloropropane
Ethylbenzene
2-Hexanone
Styrene
1,1,1,2-Tetrachloroethane
Tetrachloroethene
Xylene

1,4-Difluorobenzene
Benzene
Bromodichloromethane
Bromofluorobenzene (surrogate)
Carbon tetrachloride
2-Chloroethyl vinyl ether
1,2-Dibromoethane
Dibromomethane
1,2-Dichloroethane
1,2-Dichloroethane-d_4 (surrogate)
1,2-Dichloropropane
1,1-Dichloropropene
cis-1,3-Dichloropropene
trans-1,3-Dichloropropene
4-Methyl-2-pentanone
Toluene
Toluene-d_8 (surrogate)
1,1,2-Trichloroethane
Trichloroethene

1,2-Dichlorobenzene-d_4
Bromobenzene
n-Butylbenzene
sec-Butylbenzene
tert-Butylbenzene
2-Chlorotoluene
4-Chlorotoluene
1,2-Dibromo-3-chloropropane
1,2-Dichlorobenzene
1,3-Dichlorobenzene
1,4-Dichlorobenzene
Hexachlorobutadiene
Isopropyl benzene
p-Isopropyltoluene
Naphthalene
n-Propylbenzene
1,1,2,2-Tetrachloroethane
1,2,3-Trichlorobenzene
1,2,4-Trichlorobenzene
1,2,3-Trichloropropane
1,2,4-Trimethylbenzene
1,3,5-Trimethylbenzene

Calibration Report

Title: ID FILE FOR CAPILLARY METHOD 8260 INST. #4 WATERS
Calibrated: 930223 11:11

Compound	Files: >BE222 RF 1.00	>BD222 RF 5.00	>BF222 RF 10.00	>BC222 RF 15.00	>BG222 RF 20.00	$\overline{RF}$	% RSD	
Dichlorodifluoromethane	5.37842	4.50947	4.37612	4.23884	4.41810	4.58419	9.916	
Chloromethane	1.36942	1.18341	1.18491	1.12744	1.22996	1.21903	7.514	
Vinyl Chloride	1.85092	1.67740	1.66482	1.57720	1.76964	1.70800	6.150	
Bromomethane	2.92593	2.41279	2.50760	2.47381	2.67102	2.59823	7.952	
Chloroethane	1.31173	1.02669	1.05105	1.04007	1.08779	1.10347	10.749	
Trichlorofluoromethane	6.77075	5.74918	5.81932	5.49224	6.14974	5.99625	8.212	
1,1-Dichloroethene	2.47794	2.21027	2.19692	2.20271	2.38564	2.29470	5.640	
Methylene Chloride	2.47158	1.53525	1.86338	1.57676	1.75917	1.84123	20.469	
Acetone	-	-	-	-	-	-	- *	
Acrolein	-	-	-	-	-	-	- *	(Conc=160.0,800.0,1600.0,2400.0,3200.0)
Acrylonitrile	-	-	-	-	-	-	- *	(Conc=32.0,160.0,320.0,480.0,640.0)
trans-1,2-Dichloroethene	2.57297	2.16205	2.36502	2.28754	2.46702	2.37092	6.691	
1,1-Dichloroethane	4.65722	3.76109	3.68970	3.74524	4.13414	3.99748	10.224	
2,2-Dichloropropane	2.51400	2.31319	2.46570	2.13651	2.57333	2.40055	7.344	
cis-1,2-Dichloroethene	2.16528	1.99292	2.05956	2.10144	2.08690	2.08122	3.017	
Chloroform	4.95673	3.86776	4.18115	4.03420	4.32681	4.27333	9.790	
Bromochloromethane	1.01052	.91577	.97102	.93846	.98191	.96353	3.853	
1,1,1-Trichloroethane	3.34677	1.74551	2.00816	1.95820	2.20929	2.25359	28.087	
2-Butanone	-	-	-	-	-	-	- *	
Carbon Disulfide	-	-	-	-	-	-	- *	
Iodomethane	-	-	-	-	-	-	- *	
Vinyl Acetate	-	-	-	-	-	-	- *	
Carbon Tetrachloride	.80681	.72326	.74587	.70696	.71907	.74040	5.363	
1,2-Dichloroethane-d4	.21537	.20294	.19866	.20754	.21524	.20795	3.564	(Conc=10.0,10.0,10.0,10.0,10.0)
1,1-Dichloropropene	.69999	.58064	.60734	.57959	.58972	.61145	8.296	
Methyl-tert-Butyl Ether	-	-	-	-	-	-	- *	
Benzene	1.00397	.82777	.79289	.80507	.83522	.85298	10.095	
1,2-Dichloroethane	.30950	.24748	.23940	.24228	.25099	.25793	11.312	
Toluene-d8	.95423	.96079	.95151	.95529	.92851	.95007	1.317	(Conc=10.0,10.0,10.0,10.0,10.0)
Trichloroethene	.65306	.52552	.54122	.58433	.59422	.57967	8.639	
2-Chloroethyl vinyl ether	-	-	-	-	-	-	- *	
1,2-Dichloropropane	.38721	.32261	.30817	.32653	.31993	.33289	9.352	
Bromodichloromethane	.73317	.62153	.63334	.69409	.67591	.67161	6.777	
Dibromomethane	.25385	.20517	.22270	.23646	.22667	.22897	7.832	
4-Methyl-2-Pentanone	-	-	-	-	-	-	- *	
trans-1,3-Dichloropropene	.47348	.26118	.26741	.29550	.29888	.31929	27.494	
Toluene	1.22249	1.07188	1.05335	1.06495	1.07890	1.09831	6.378	
1,2-Dibromoethane	.29207	.25517	.26933	.27205	.27160	.27204	4.848	
cis-1,3-Dichloropropene	.47348	.36855	.36660	.36966	.36563	.38879	12.185	
1,1,2-Trichloroethane	.21691	.15480	.16007	.17179	.16098	.17291	14.666	
Tetrahydrofuran	2.00616	.42129	.19953	.13872	.10317	.57377	141.207	

* SINGLE POINT CALIBRATION ONLY ON THESE COMPOUNDS
MGK 3/25/93

RF - Response Factor (Subscript is amount in ppb)

$\overline{RF}$ - Average Response Factor

%RSD - Percent Relative Standard Deviation

Calibration Report

Title: ID FILE FOR CAPILLARY METHOD 8260 INST. #4 WATERS
Calibrated: 930223 11:11

Compound	Files: >BE222 RF 1.00	>BD222 RF 5.00	>BF222 RF 10.00	>BC222 RF 15.00	>BG222 RF 20.00	$\overline{RF}$	% RSD	
Bis(Chloromethyl)Ether	-	~~.09849~~	~~.00634~~	~~.00694~~	~~.00862~~	~~.03010~~	~~151.522~~	MH 4126193
2-Hexanone	-	-	-	-	-	-	- *	
Ethyl methacrylate	-	-	-	-	-	-	- *	
Tetrachloroethene	1.25785	1.17558	1.17726	1.06277	1.21355	1.17740	6.143	
1,3-Dichloropropane	.44079	.34413	.34233	.35389	.37485	.37120	11.043	
Dibromochloromethane	.63584	.56499	.57237	.56043	.61955	.59064	5.855	
Chlorobenzene	1.08077	1.00905	1.03010	.98636	1.06220	1.03369	3.712	
1,1,1,2-Tetrachloroethane	.59445	.57340	.61700	.59486	.63184	.60231	3.750	
Ethylbenzene	.53700	.49772	.52730	.48600	.55108	.51982	5.233	
p-Xylene	3.47138	2.92775	3.07390	2.91489	3.18103	3.11379	7.326	
m-Xylene	3.47138	2.92775	3.07390	2.91489	3.18103	3.11379	7.326	
o-Xylene	1.65886	1.41318	1.42318	1.36836	1.48061	1.46884	7.727	
Styrene	.91782	.78798	.84495	.84996	.89991	.86012	5.943	
Bromoform	.34144	.32258	.37364	.39524	.40437	.36746	9.489	
Bromofluorobenzene	-	-	-	-	-	-	-	(Conc=10.0,10.0,10.0,10.0,10.0)
Isopropylbenzene	3.39053	3.03859	2.70147	2.43668	2.58829	2.83111	13.535	
1,1,2,2-Tetrachloroethane	.44862	.34875	.31118	.30990	.30216	.34412	17.770	
Bromobenzene	.85184	.84059	.76152	.71543	.69822	.77352	9.100	
1,2,3-Trichloropropane	.08741	.07597	.08176	.07740	.07456	.07942	6.570	
n-Propylbenzene	.83413	.75008	.65228	.59358	.62850	.69171	14.251	
2-Chlorotoluene	.81392	.67540	.58869	.54141	.56386	.63666	17.490	
trans-1,4-dichloro-2-butene	-	-	-	-	-	-	- *	
4-Chlorotoluene	.72944	.69461	.58517	.57015	.53603	.62308	13.487	
1,3,5-Trimethylbenzene	2.87208	2.36642	2.10034	1.93220	1.99522	2.25325	17.027	
tert-Butylbenzene	3.01161	2.58871	2.29350	2.09536	2.22264	2.44237	14.992	
1,2,4-Trimethylbenzene	2.56373	2.26988	1.97234	1.83816	1.89142	2.10711	14.473	
sec-Butylbenzene	4.33022	3.63201	3.18812	2.93356	3.10366	3.43751	16.343	
p-Isopropyltoluene	3.47296	2.89590	2.55735	2.27457	2.41853	2.72386	17.545	
1,3-Dichlorobenzene	1.62857	1.33461	1.21593	1.12984	1.16030	1.29385	15.674	
1,4-Dichlorobenzene	1.75171	1.19783	1.28061	1.21353	1.17500	1.32374	18.316	
1,2-Dichlorobenzene	1.35580	1.14418	.99143	.93222	.90635	1.06600	17.490	
n-Butylbenzene	3.58014	2.87226	2.56712	2.33564	2.41956	2.75494	18.314	
1,2-Dibromo-3-Chloropropane	.03631	.06206	.07587	.07170	.05291	.05977	26.509	
1,2,4-Trichlorobenzene	1.18069	.84326	.71392	.76119	.67914	.83564	24.229	
Hexachlorobutadiene	2.86520	1.90771	1.67087	1.49922	1.54933	1.89847	29.654	
Naphthalene	.61348	.46555	.37663	.42959	.36196	.44944	22.393	
1,2,3-Trichlorobenzene	.98157	.64452	.48334	.55581	.47660	.62837	33.224	

RF - Response Factor (Subscript is amount in ppb)

$\overline{RF}$ - Average Response Factor

%RSD - Percent Relative Standard Deviation

APPENDIX C.

Log Book Example

Tune Form Examples

Sample Tracking Sheet Example

GC/MS VOLATILE ANALYSIS LOG - 1990

WESTON

INSTRUMENT ID # 4 CLAS ID # 21

ANALYSIS DATE TIME 12/2&3/91	FILE NAME	RFW NUMBER YYMM G XXX / XXX	CLIENT / SAMPLE ID	SPARG NO.	PREPARATION SAMPLE WT/VOL	PREPARATION INJ. VOL. (ul)	INST DIL.	ANALYSIS STATUS XS	ANALYSIS STATUS LIST	ANALYSIS STATUS DEL	COMMENTS	SECTORS	TAPE NO.	ANAL INIT
1345	AAC28	BFB Tune	Tune	NA	5ml OFW						+2ul BFB #136	29		K Beamish
1345	BAC28	10 Std	Std		25ml OFW						+5ul 144 +10ul 145	377		
1519	CAC28	91GVE392	MB1 Blank								+ 10 ul 12 hr 5ul 144	151		
1613	OSH29	91126580	020 TRC Environ.		25ml							189		
1701	OSH30		021									104		
1745	OSH31		022									157		
1828	CBC28	91GVE393	MB1S Spike Blk		25ml OFW						+10ul 142	164		
1911	OSH32	91126580	020 TRC Environ.	NA	25 ml						+5ul ISS 144	192		K Beamish
12/30/91 0828	AAC30	BFB TUNE	TUNE		5ml OFW						2uL BFB 136	30		DEKochlert
0912	BAC30	STD 10	STD		20ml OFW					139	4uL ISS 4uL 137, 138	241		
1003	CAC30	91GVE393	MB1 BLANK								4uL ISS	125		
1052	OSR01	9112G627	001 ARCS		20mL SAMP							193		
10:40 AM 1135	12/30/91 OSR02	Data Collected	002 Time Check									126		
1229	OSR03		003									122		

Analyst Signature/Date DEKochlert 12/30/91 Reviewed by Signature/Date Jennifer S. O'Brien 12-30-91

BLANK FORM USED TO "LOG IN" SAMPLES TO VOLATILE DEPARTMENT

GCL #	CLIENT	FILE NAME(S)	DIL.	TUNE NAME	SPECIAL INFO
		(NEXT FILE NAME AVAILABLE IN FILE NAME LOGBOOK)			

QUAL475	TAT 0674 (1) OLD AMTK				
001	soi /	ORN01		KAC18	OK
002		ORN02 ORN09		KAC18 KAC18	ISSC 1/2 SO Redo OK
003		ORN03		KAC18	OK
004		ORN04		KAC18	OK
005		ORN05 S ORN07 T ORN08		KAC18	OK
006		ORN06		KAC18	OK

EXAMPLE OF A COMPLETED TRACKING RECORD

BLANK TUNE FILE FORM USED TO TRACK A 12 HOUR BATCH ON ONE INSTRUMENT

TUNE RUN NAME (ascii)	TUNE RUN TIME GOOD UNTIL	TUNE FILE: STO RUN NAME: ID FILE :	TUNE?
FORM6 :	FORM7 :	QCAL: ID_ :	STO/CLP: STO AREA :

GEL # CLIENT	FILE NAME (ascii, PC)	MISCELLANEOUS	MODEL ID	L 5	L 6	L 7	MATRIX CLIENT
1							
2							
3							
4							
5							
6							
7							
8							
9							
10							
11							
12							
13							
14							
15							
16							
17							
18							

SPREADSHEETS	TEST	RFW BATCH #	PC	MISCELLANEOUS

[redacted]

A COMPLETED TUNE FILE FORM

TUNE RUN NAME (ascii): eKAC18 .LV

TUNE RUN TIME GOOD UNTIL: 0811

TUNE FILE: STD RUN NAME: e TUNE3 LAC18 .C

ID FILE :

FORM6 : ✓ FORM7 : ✓ IQCAL: ID_mS3: ✓ :STD/CLP: STD APER : 25441

	GEL # CLIENT		FILE NAME (ascii, PC)	MISCELLANEOUS	MODEL 1	2	3	4	5	MATRIX 6	7
1	91GVC392-mB1	C	mAC18 .LV	OK							
2	mB1S	C	MBC18 .LV	OK							
3											
4	BP 9112G407-001	C	ORG03 .LV	1/200 Acclo OK						So	624X
5	BP 9112G447-001	C	ORJ07 .LV	1/2000 ACRYLO, ACETO, ACE EDIT HEADER OK							
6	003	C	ORJ08 .LV	1/20,000 Acrolein, Acelo Redo 1/250,000 Acelo							
7	TAT 9112G475-001	C	ORN01 .LV	OK						S	H/CLP
8	002	C	ORN02 —	155 1/2 So Redo							
9	003	C	ORN03 .LV	OK							
10	004	C	ORN04 .LV	OK							
11	005	C	ORN05 .LV	OK							
12	006	C	ORN06 .LV	OK							
13	0055	C	ORN07 .LV	OK							
14	005T	C	ORN08 .LV	ISSUE OK							
15	002	C	ORN09 .LV	OK							
16	91126447-003	C	ORJ09 1/250,000 .LV	4ml at 2500 1/200 OK							
17											
18											

SPREADSHEETS	TEST	RFW BATCH #	PC	MISCELLANEOUS
GVC392	X	447	A	up'd ✓

[redacted]

APPENDIX D.

Review Form

REVIEW FORM

WESTON/Gulf Coast Laboratories, Inc.
GC/MS Data Review Checklist

Client:_____ RFW#:_____ Test:_____
Methods:_____

Date:_____ Reviewer:_____ (First Review)
Date:_____ Reviewer:_____ (Second Review)

Form 1/ Associated Data Hold Time Met ____ pH Check ____
Run Log ____ Archive Log ____

Reviewer 1	Reviewer 2	
___	___	Review total Ion Chromatogram for obvious leaks and/or other unacceptable phenomena. Confirm re-analysis if required.
___	___	Check header for correctness (correct tune file, RFW number, extraction volume, final volume, dilution factor, calibration of appropriate ID file, data and time of analysis within tune time).
___	___	Confirm presence of background subtracted spectra for each TC (target compound) reported.
___	___	Confirm that all appropriate "E" values are present. Confirm that dilutions are reported for only those compounds with an "E" value.
___	___	Confirm that the RFW# on the data file header is the same as the one on the Form 1.
___	___	Confirm that each TC to be reported appears on the Form 1. Manually calculate one concentration. Confirm that those to be flagged with a B, J, or E have been. Confirm correct matrix, target list and method of reporting (wet/dry).
___	___	Review TIC (tentatively identified compound) identification and retention time. Manually calculate one concentration.

Comments: (Provide explanation for samples reported past hold time with no initial good analysis.)

Form 2/Associated Data

___	___	Confirm correct matrix.
___	___	Confirm that all QC and samples for the RFW# batch are represented.
___	___	Confirm all surrogates are within control limits. Confirm re-analysis for any outliers and flag for documentation in the case narrative.

Comments:

Form 3/Associated Data

___	___	LCS's (BS/BSD): The department will be generating control limits from real analyses. At present, these recoveries are not used to determine re-analysis or re-extraction of the extraction batch.
___	___	Confirm proper batch number.
___	___	Review data and investigate any obvious problems (recoveries low or high etc...). Flag outliers for the case narrative.
___	___	Confirm Matrix

Reviewer 1 | Reviewer 2

Matrix Spikes/Duplicates:

__ __ Confirm that matrix spike data correlates with the unspiked analysis.
__ __ Review data and investigate any obvious problems (recoveries low or high, etc...). Flag outliers for the case narrative.
__ __ Correct matrix.

__ __ Confirm the presence of LCS data for every data package (except those X-TCL tests that do not include any of the spike compounds). Confirm the presence of the MS/MSD for the chosen RFW# or client.

Comments:

Form 4/Associated Data

__ __ Correct Matrix
__ __ Confirm proper extraction batch number, correct associated data files and data.
__ __ Confirm absence of any TCL or if present 1) <PQL or 2) <5x PQL for allowed compounds.
__ __ Confirm all RFW's in the batch/package appear on the appropriate Form 4.

Comments:

Form 5/Associated Data

__ __ Confirm correct tune data file, data and time.
__ __ Confirm abundances are within control limits.
__ __ Confirm presence of each sample, standard, method blank, LCS and matrix spike.
__ __ Review correctness of data and time of analysis for obvious errors.
__ __ List any samples anlayzed past tune time.

Comments:

Form 6,7/Associated Data

__ __ Confirm presence of Forms 6 & 7. Confirm presence of appropriate initial calibration date files for CLP packages.
__ __ Confirm correct standard data file and initial calibration date on Form 7.
__ __ Confirm control limits were met on continuing calibration, if required.
__ __ Confirm presence of initial calibration data for extra compounds (CBRPT, CBCHK) if required.

Comments:

Form 8 (For CLP Packages)

__ __ Confirm that all areas and retention times are within limits. Flag outliers for the case narrative.

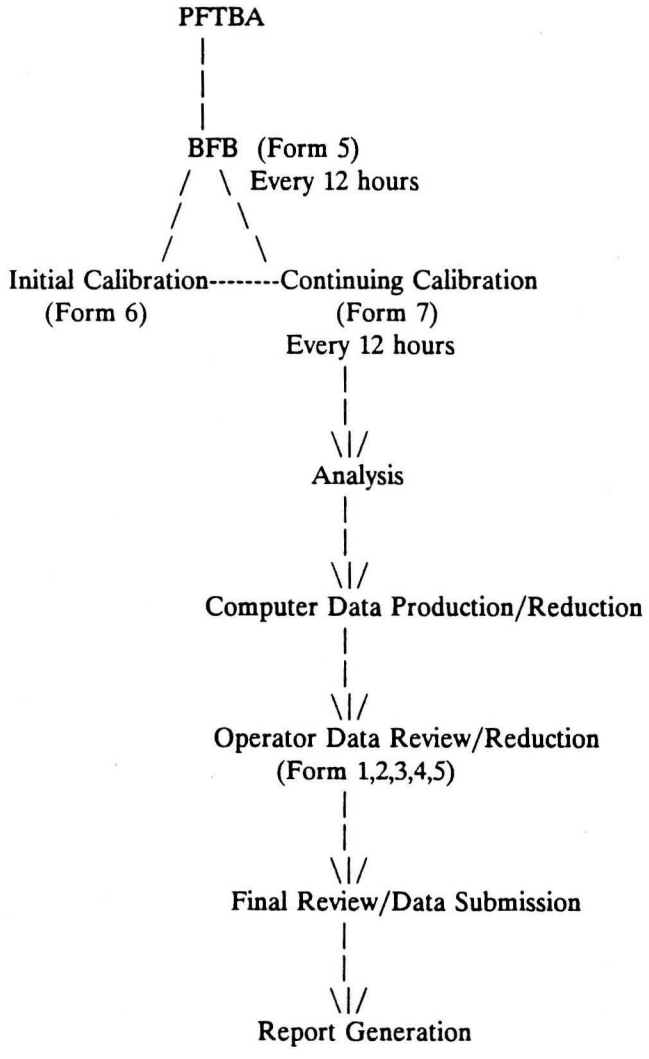
Comments:

Appendix E.

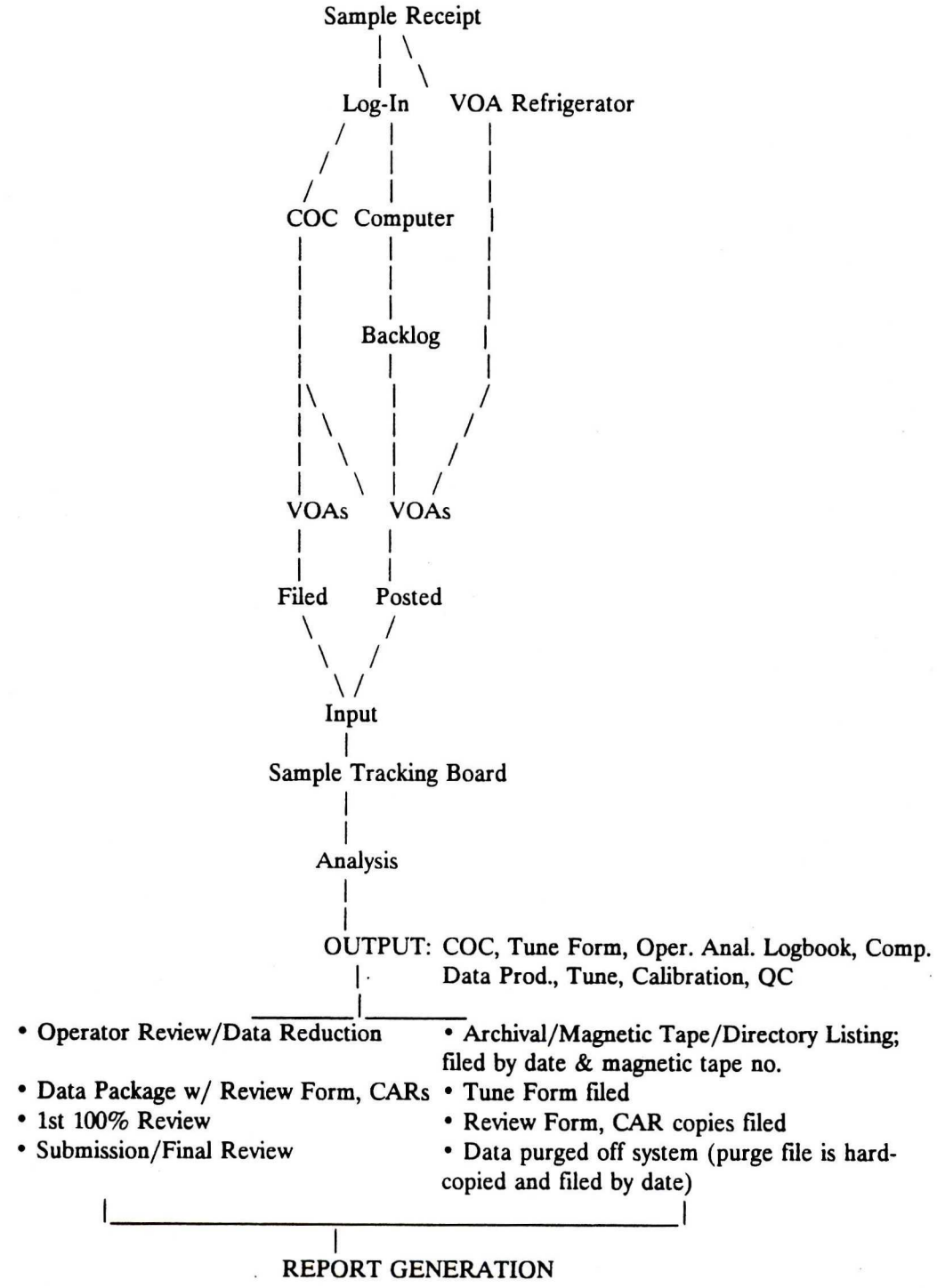
Analysis and Sample Tracking Flowcharts

ANALYSIS SCHEME FLOWCHART

(Terms defined in the Section 9)



Sample Tracking Flowchart (for EACH unique RFW batch #)



ORGANIC ANALYSIS PROTOCOL
Gas Chromatography/Mass Spectrometry (GC/MS)
Analysis of Volatile Organic Compounds by Method 8260

These Approval Signatures Are Kept on File
with WESTON®'s Analytics Division
QA Standard Practice Records

REVISION NUMBER: 01

	Printed Name:	Signature/Date:
Written By:	Marilyn G. Krueding GC/MS Unit Leader	Marilyn G Krueding 4/26/93
Approved By:	Jeff A. Kaczinksi GC/MS Section Manager	Jeff A. Kaczinski 4-26-93

Historical File: Revision 00: 06/22/92
Revision 01: 04/26/93

Reasons for Change, Revision 01:

- Changes in surrogate, internal standard spiking, and the working 5-point standard solutions;
- BFB tune with 25 ng, rather than 50 ng;
- Figure 4 and Attachment B updated.

Eff. Date: 03/19/93 Initiated By: QC Department Approved By: S. S. Iyer Authorized By: A. M. Henry SP No. 21-15G-245.1

S. Iyer

INORGANIC ANALYSIS PROTOCOL
Mercury Analysis by Leeman AutoAnalyzer
EPA Methods 245.1; SW-846 Method 7470/7470; USEPA Document No. ILM02.1

CONTROLLED DISTRIBUTION
COPY # : Uncontrolled
ISSUED TO : Tech Mgr QAP (VHI)
Full Signature Approvals Are Kept on File
with WESTON®'s Analytics Division
QA Standard Practice Records

REVISION NUMBER: 02

1.0 PURPOSE

Determination of mercury in aqueous and nonaqueous media.

2.0 REFERENCE

This SOP was written using EPA 600/4-79-020 Method 245.1; SW-846, 3rd Edition Revision 0, Methods 7470/7471; USEPA Document ILM02.1 as references.

3.0 METHOD SUMMARY

The flameless AA procedure is a physical method based on the absorption of radiation at 253.7 nm by mercury vapor. The mercury is reduced to the elemental state and swept from solution and passed through a cell of a double beam AA. Absorbance is a function of mercury concentration.

4.0 INTERFERENCES

Chloride, sulfide, certain volatile organic materials.

5.0 SAMPLE COLLECTION PRESERVATION AND HANDLING

Acidify to pH <2 at the time of collection. Holding time is 28 days from collection for glass containers, 24 days from collection for plastic containers.

6.0 PREVENTIVE MAINTENANCE

The PS200 requires some routine daily maintenance as well as some scheduled and non-scheduled periodic maintenance. All maintenance will be recorded in the instruments maintenance logbook. The following maintenance schedule lists the various maintenance procedures and when they should be performed. Each of these procedures is described in the following sections.

6.1 Maintenance Schedule

Equipment	Schedule
Drying Tube	Must be Changed Daily!!
Pump Tubing	Weekly, or as needed
Lamp	Replace as needed (avg 4 mos. - 1 yr.)
Optical Cell	Clean as needed (typically monthly)
Liquid Gas Separator	Replace every 1-3 yrs, as needed
Internal Tubing	Should not require replacement under normal circumstances

6.2 Packing and Changing the Drying Tube

Under normal use, the drying tube on the PS200 must be changed each morning before you run samples. (The drying tube is located on the front panel on the left side of the PS200) You may wish to pack several tubes at one time and store them in an airtight container so that you have a ready supply.

To pack a tube, plug one end with quartz wool, pour in magnesium perchlorate to fill tube, and plug the other end with quartz wool.

To change a tube, slightly loosen the nuts that hold the tube in at either end and slide the used tube out of the fittings. Slide a fresh tube into the fittings and tighten the fittings with your fingers to make a gas-tight seal.

To clean a tube, remove the quartz wool and the magnesium perchlorate. Either dispose of as a solid waste or dissolve in water and dispose of as a liquid waste. Clean the tube with ordinary laboratory glassware cleaner and dry thoroughly.

6.3 Replacing and Exercising Pump Tubing

Pump Tubing should be replaced weekly or when it shows signs of wear. There are four pump tubes: two for drainage, one for sample, and one for reductant. Each tube is fed through a pump cassette which then clamps onto the pump head. Slide a tube through the plastic clips at the bottom of a cassette until the plastic tab is secure. Hold the tube taut, slide the loaded cassette onto the pump head, and lock the clamp up. Repeat for the remaining tubes, then connect the tubes ends.

For optimal performance, run DI water through new tubes for one hour to exercise them before using them for running samples. To do this, select INSTRUMENT from the Main Menu and then select OPERATION. The INSTRUMENT:OPERATION screen will appear. Set the Pump Rate flow to the standard rate for 5 mL/min (Type R and M and 5 Enter). Wait for one hour and then connect the tubing to the appropriate fluids.

NOTE: This procedure only needs to be done once, when the tubes are new and unused.

6.4 Replacing the Lamp

The mercury lamp has a life of about 2000 hours, between four months and a year of use. The lamp needs to be replaced if the relative absorbance of a standard has changed significantly while the optical cell is clean. If the lamp is suspected, it is faster to replace the lamp and recalibrate than to clean the optical cell.

NOTE: Before installation, clean the new lamp quartz with methanol and wipe it dry. Do not get finger prints on the lamp and do not face the printing on the lamp toward the optical cell.

To replace the lamp;

6.4.1 Turn off the lamp (press the blue button on the front of the PS200).

6.4.2 Remove the front panel for the PS200 (lift up and out).

6.4.3 Remove the optical assembly.

6.4.4 Remove the two screws on the lamp housing and take off the lamp cover.

6.4.5 Twist the lamp 90° and slide it straight out.

6.4.6 Insert the new lamp and rotate it 90° in the reverse direction to secure it in place. Make sure that the lettering on the lamp will be facing to the left of the instrument when it had been reinstalled into the PS200. If it is not, remove the lamp and reinsert it correctly.

6.4.7 Replace the optical assembly.

6.5 Cleaning the Optical Cell

If the relative absorbance of standards differs significantly from that of previous calibrations, the optical cell (located inside the front panel) may be dirty and must be cleaned:

· Turn the lamp and the PS200 power off and remove the front panel by lifting it up and out.

· Remove the optics clamps, disconnect the detector, and rotate and lift out the assembly. Disconnect the gas lines.

· Remove the six screws holding the lamp spacer and the detector spacer onto the optical cell.

· Inspect the two ends with the lenses. If the external surface of the lenses appear to be the only contaminant, then clean. To clean use methanol. Install if no other cleaning is necessary.

· Disassemble the optical cell (using the allen wrench provided on the inside of the front cover) by removing (in order) the screws, lens, and gasket at each end.

· Carefully clean the inside of the cell with laboratory glassware cleaner, taking care not to scratch the inside surfaces. Rinse thoroughly, first with water and then with DI water. Dry the cell in the oven (free of contaminants) for one hour at approximately 40 - 50°C.

· Clean the lenses with laboratory glassware cleaner and rinse thoroughly with hot tap water. Flush lightly with methanol and dry by air or vacuum oven (maximum 50°C).

· Replace the gaskets (this is recommended although not required unless the gasket shows signs of wear) and reassemble the optical cell. Cleaning of the gaskets should only be done with DI water.

6.6 Replacing the Liquid Gas Separator

The liquid gas separator (transparent block on the chemical panel of the PS200) should only need to be replaced once every one to three years, depending on the amount of use it receives.

To replace the separator, shut off the gas and liquid flow and flush the tubing with DI Water for safety purposes. Disconnect the four lines and remove the two screws. Remove the unit from the system, screw on a new one, reconnect the four lines, and turn the gas and liquid flow back on.

6.7 Replacing Internal Tubing

Internal gas and teflon tubes should last indefinitely and should not need to be replaced. Periodically inspect all tubing for restrictions or blockages. If tubing should need to be replaced, do so one piece at time to avoid any confusion while making connections.

7.0 INSTRUMENTATION AND EQUIPMENT

Instrument: Leeman Labs Model PS200 Automated Mercury Analyzer

8.0 STANDARDS, REAGENTS AND QC SOLUTIONS

8.1 Standard Stock Solution I; 1000 ppm

A 1000 ug/mL concentrated mercury standard is purchased from an outside supplier. The concentrated standard expires one year from the date of receipt. This concentrated standard is diluted down to a working range on a daily basis.

- Life of Reagent: one year
- Storage Requirements: none

8.2 Working Standard Solution I; 100 ppb

To a 1.0 L volumetric flask filled with ~800 mL DI water, transfer 100 uL of Stock Solution I to the flask using a 100 uL Eppendorf pipette. Add 2.5 mL conc. nitric acid as a preservative. Dilute to volume with DI Water. Invert and mix to insure complete mixture.

*For use in spiking Matrix Spikes, CRAs & the Standard Curve.

- Life of Reagent: 24 hours
- Storage Requirements: none

8.3 Standard Stock Solution II; 1000 ppm

Purchased from an outside supplier as a 1000 ppm solution and is from an alternate source than the standards.

- Life of Reagent: one year
- Storage Requirements: none

8.4 Working Standard Solution II; 200 ppb

To a 1.0 L volumetric flask filled with ~800 mL DI water, add 2.5 mL concentrated nitric acid (as a preservative) and 200 uL of Standard Stock Solution II to the flask (using a 100 uL Eppendorf pipette). Dilute to volume with DI water and invert several times to mix.

*For use in spiking the ICV/CCV and LCS/LCSD.

- Life of Reagent: 24 hours
- Storage Requirements: none

8.5 Sodium Chloride-Hydroxylamine Hydrochloride Solution

Prepare by dissolving 240 g of sodium chloride and 240 g of hydroxylamine hydrochloride in sufficient deionized water to make 2000 mL of solution.

- Life of Reagent: one year
- Storage Requirements: none

8.6 Stannous Chloride Solution

Prepare by dissolving 10 g of stannous chloride in 10% hydrochloric acid to make 100 mL of solution.

- Life of Reagent: one month
- Storage Requirements: none

8.7 Potassium Permanganate, 5%

Prepare by dissolving 175 g of potassium permanganate into 3500 mL of DI water.

- Life of Reagent: one year
- Storage Requirements: none

8.8 Potassium Persulfate, 5%

Prepared by dissolving 175 g of potassium persulfate into 3500 mL of DI water.

- Life of Reagent: one year
- Storage Requirements: none

8.9 Miscellaneous Reagents:

Hydrochloric Acid, Conc. (soil only)
Nitric Acid, Conc.
Sulfuric Acid, Conc. (aqueous only)

8.10 DI Water Type II

9.0 PROCEDURE

9.1 Optimum Concentration Range

0.2 ug/L - 5 ug/L

9.2 Instrument Detection Limit

Approximately 0.02 ug/L.

9.3 Sample Preparation

9.3.1 Merucry Water Digestion Procedure - EPA Method 245.1/ILM02.1

Action	Full Scale
Sample Volume	100 mL
Reaction Vessel	BOD Bottle, 300 mL
Sulfuric Acid (conc.)	5 mL
Nitric Acid (conc.)	2.5 mL
Potassium Permanganate, 5% Sol. (W/V)	15 mL
Potassium Persulfate, 5% Sol. (W/V)	8 mL
Preparation	Water Bath, 2 hrs. @ 90 - 95°C, Cool
Hydroxylamine Addition	6 mL
Total Volume	136.5 mL

Proceed with the Stannous Chloride addition.

9.3.2 Merucry Water Digestion Procedure - SW-846 Method 7470

Action	Midi Scale	Full Scale
Sample Volume	33 mL	100 mL
Reaction Vessel	Screw cap 25 x 200 mm, 75 mL capacity	BOD Bottle, 300 mL
Sulfuric Acid (conc.)	1.67 mL	5 mL
Nitric Acid (conc.)	0.83 mL	2.5 mL
Potassium Permanganante, 5% Sol. (W/V)	5 mL	15 mL
Potasssium Persulfate, 5% Sol. (W/V)	2.67 mL	8 mL
Preparation	Water Bath, 2 Hrs. @ 90 - 95°C, Cool	Water Bath, 2 Hrs. @ 90 - 95°C, Cool
Hydroxylamine Addition	2 mL	6 mL
Total Volume	45 mL	136.5 mL

Proceed with the Stannous Chloride addition.

9.3.3 Mercury Soil Digestion Procedure - SW-846 Method 7471

Action	Midi Scale	Full Scale
Sample Weight	0.1 gram	0.2 - 0.3 grams
Reaction Vessel	Scre cap 25 x 200 mm, 75 mL capacity	BOD Bottle, 300 mL
DI Water, Type II	2.5 mL	5 mL
Aqua Regia [3:1 HCl (conc.) to HNO_3 (conc.)]	2.5 mL	5 mL
Preparation	Water Bath, 2 min. @ 90 - 95°C, Cool	Water Bath, 2 min. @ 90 - 95°C, Cool
DI Water, Type II	25 mL	50 mL
Potassium Permanganate, 5% Sol. (W/V)	7.5 mL	15 mL
Preparation	Water Bath, 2 min. @ 90 - 95°C, Cool	Water Bath, 30 min. @ 90 - 95°C, Cool
Hydroxylamine Addition	3 mL	6 mL
Total Volume	40.5 mL	Dilute to 100 mL

Proceed with the Stannous Chloride addition.

9.3.4 Merucry Water Digestion Procedure - EPA Methopd 245.5/ILM02.1

Action	**Full Scale**
Sample weight	0.2 - 0.3 grams
Reaction Vessel	BOD bottle, 300 mL
Sulfuric Acid (conc.)	5 mL
Nitric Acid (conc.)	2.5 mL
Preparation	Water Bath, 2 min. @ 90 -95°C, Cool
DI Water, Type II	50 mL
Potassium Permanganate, 5% Sol. (W/V)	15 mL
Potassium Persulfate, 5% Sol. (W/V)	8 mL
Preparation	Water Bath, 30 min. @ 90 - 95°, Cool
Hydroxylamine Addition	6 mL
Total Volume	Dilute to 100 mL

Proceed with the Stannous Chloride addition.

9.4 Working Calibration Standards for Mercury in Water

Standard (ug/L)	mL of Stock Soln.	Stock Conc. (ug/L)	Final Volume (mL)
Blank	0.0	----	100
0.2	0.2	1000	100
0.5	0.5	1000	100
1.0	1.0	1000	100
3.0	3.0	1000	100
5.0	5.0	1000	100
ICV/CCV (2.0 ug/L)	1.0	2000	100

9.5 Preparing the System

9.5.1 The following procedures must be performed each morning before warming up the system:

9.5.2 Press the F10 macro key to stop any currently running macro.

9.5.3 Change the drying tube. Refer to maintenance, Section 6.0 for instructions.

9.5.4 Release the clamps and check the pump tubing for wear. Under normal use, the tubes will need to be replaced once a week. To replace the tubing, refer to maintenance, Section 6.0 for instructions.

9.5.5 Check the reductant volume and refresh, if needed.

9.5.6 Clean the rinse tank using standard lab cleaning practices, add fresh rinse.

9.5.7 If the lamp has been off then turn on the lamp power and allow the lamp to warm up for at least 45 minutes.

9.5.8 If the system is shut off, power up all components and perform COLDSTRT macro.

9.5.9 You are now ready to start up the system.

9.6 Start-up Procedures

9.6.1 The start-up routine you will use depends on the current state of the system. If it is in Overnite mode, use the Warmstart macro (Section 9.7). If the system has been completely powered down, you will need to run the Coldstart macro instead (Section 9.8).

9.7 Warm Start

9.7.1 The Warmstart macro is used to prepare the PS200 for operation if it is being started up from a short-term (overnight) shutdown.

9.7.2 To run the Warmstart macro, press the F2 macro key on the keyboard. Type WARMSTRT and press ENTER. The system will wait for several minutes and then turn on the pump and the gas flow to protocol speed. When the system is stable, a beep will sound and an "Operation Complete" message will appear on the screen. The PS200 is now ready for operation.

9.8 Cold Start

9.8.1 The Coldstart macro procedure is used to prepare the PS200 for operation if the system has been shut down for an extended period of time. This procedure turns on the liquid and the gas flow and then waits until the system thermally equilibrates before beeping to indicate that it is ready to run. You should then perform an aperture test and make any necessary adjustments to the aperture before you run samples.

9.8.2 To run the Coldstart macro, press the F2 macro key on the keyboard, type COLDSTRT and press ENTER. The Coldstart procedure takes approximately 2 1/2 hours. Do not attempt to operate the PS200 before this procedure is complete, or its performance will be significantly impaired.

9.8.3 When a beep has sounded and an "Operation Complete" message is visible on the screen, indicating the completion of the Coldstart procedure, you must check the apertures on the optical cell and make any necessary adjustments; this procedure is documented in Section 2.10, steps 1 and 2 of the operator's manual. When the aperture adjustments are completed, the PS200 is ready for operation.

9.9 Software Setup

9.9.1 In order to run samples, you must enter all necessary information regarding the protocol, sample ID's, calibration values, and autosampler parameters into the software. This information is entered into a series of screens which are accessed from the Main Menu. (You can display the Main Menu at any time by pressing the F1 key on your keyboard.)

9.9.2 Perform each of the following steps in sequence to set up the software. When you have completed these steps, the PS200 will be able to run samples automatically.

NOTE: The steps below comprise the basic daily software setup sequence. The PS200 software also contains numerous advanced functions. Refer to the PS Series Reference Guide for a detailed description of the many other keys and functions available for use with this system.

9.9.3 Name the Protocol: Protocols are operational determinations (parameters) for running calibrations and samples. You must name the desired protocol to instruct the PS200 what its normal operational values will be for running the next batch of samples.

· From the Main Menu, select PROTOCOL and then select GET. The Protocol screen will appear a "Get protocol name:" message will be displayed at the bottom of the screen.

· Type the protocol name and press ENTER. This creates a protocol file.

· Press the F1 key to return to the Main Menu.

9.9.4 Name the Folder:

Once you have named the protocol, you must create a folder to hold all data generated from each sequence of operation.

· From the Main Menu, select DATA OUTPUT and then select Open folder. The Folder maintenance screen appears and an "Enter folder name:" message will be displayed at the bottom of the screen.

- Type a folder name and press ENTER. The folder is created.

- Press the F1 key to return to the Main Menu.

9.9.5 Verify Values and Integration Times

You must now check to make sure that all values and integration times are correct for running the samples:

- From the Main Menu, select PROTOCOL, then select SET Values. The Set Values screen appears.

- For normal operation, enter the following values (as Illustrated below):

Number of Integrations:	1
Uptake time	10
Weight	N
Dilution	N
Percent Recovery	N

- Press F1 to return to the Main Menu.

9.9.6 Enter values for on/offs, times, and gains

- From the Main Menu, select PROTOCOL, then select ON/OFFS, TIMES, GAINS. The on/offs, times, gains screen appears and an "Enter integration time:" message is displayed at the bottom of the screen.

- Type the desired integration time from between 1 and 30 seconds (the typically selected value is 10 seconds) and press ENTER.

- Press the F1 key to return to the Main Menu.

9.9.7 Enter the Calibration Standard Concentrations:

- From the Main Menu, in sequence, select CALIBRATION, STANDARDS, and then UNITS. The Units screen appears an "Enter units:" prompt is displayed at the bottom of the screen.

· Type the desired unit of measurement (e.g., ppb) and press ENTER. Your entry will appear in the Units column above.

· Using the hot key, select each standard on the screen (S1-S6) and enter the appropriate calibration standard concentration (e.g., S1-.00000, S2-.20000, S3-.50000, S4-1.0000, S5-3.0000, S6-5.0000)

NOTE: Do not be concerned with the UI (Update Intercept) and US (Update Slope) columns at this time. If you want more information of these fields, refer to your PS Series Reference Guide.

· Press the F1 key to return to the Main Menu.

9.9.8 Reset the Calibration Intensity Data:

· From the Main Menu, select CALIBRATION, RESET, and NEW CALIBRATION RESET. The Reset screen appears at the bottom of the screen.

· To erase any calibration data that may have already been done with this protocol, enter Y and press ENTER. An "All Date Reset" message will appear when the process is complete. (To escape this procedure, enter N instead.)

· Press the F1 key to return to the Main Menu.

9.9.9 Set the Autosampler Rinse Time:

· From the Main Menu, select AUTOSAMPLER, SETUP, and RINSE TIME (seconds). The Setup screen appears and an "Enter rinse time:" message is displayed at the bottom of the screen.

· Type the desired value in seconds (typically 50) and press ENTER.

· Press the F1 key to return to the Main Menu.

9.9.10 Set up the Racks:

· From the Main Menu, select AUTOSAMPLER and then RACK ENTRY. The Rack screen appears and an "Enter rack name:" message is displayed at the bottom of the screen.

· Type a rack name (either new or existing) and press ENTER. (If you enter a new name, you will be asked if you want to create a new rack: answer Y.)

· Fill the sample cups to be used to within 1/4" from the top (to allow for two runs). Using the autosampler layout in as a guide, load each sample cup into the rack and enter the sample ID into the appropriate (cup) position on the rack entry screen.

NOTE: For details on the INSERT key, rack calculation options, and advanced editing options, refer to the PS Series Reference Guide.

· It is important to remember that the PS200 can run two complete racks unattended.

· Press the F1 key to return to the Main Menu.

9.9.11 Define start-to finish sample sequence:

· From the Main Menu, select AUTOSAMPLER and then SETUP. Type the rack number to be run (1 or 2). The prompt "Enter rack name" is displayed at the bottom of the screen.

· Type the rack name and press ENTER. The Setup screen for that rack will appear and a "Begin cup:" prompt will be displayed at the bottom of the screen.

· Enter the number (cup position) of the first cup to be sampled and press ENTER. An "End cup:" prompt will now be displayed at the bottom of the screen.

· Enter the number of the last cup to be sampled and press ENTER.

- Press the F1 key to return to the Main Menu.

- If you are using a second rack, repeat steps 1-5.

9.10 Calibrating the System

The PS200 must be calibrated before you can run samples:

To perform a standard EPA (Method 7470) calibration, press the F2 macro key and "Macro:" prompt appears at the top of the, type AUTOCLP and press enter. The calibration routine will begin running. It is assumed that the five standards (0, 0.2, 0.5, 1.0, 3.0, and 5.0 ppb) have been loaded as standards 1 through 6. After the standards run, the check standards will run automatically. AUTOCLP will accept the calibration. "Macro:" RUNSTD will run standards only.

To perform a calibration other than a standard EPA procedure, press the STD F6 action key. The Standard screen appears and a "Run standard: 1 2 3 4 5 6 " message is displayed at the bottom of the screen. Enter the number of the standard to be run (1-6) and press enter. A "from replicate: 1 to: _ "message will then be displayed at the bottom of the screen. Enter the first number in the "from replicate:" field and last number in the "to:" field. Press ENTER. The system will run the standards.

NOTE: To stop a procedure at any time, press the Stop F10 action key.

The results of the calibration are automatically stored. To review the results, select CALIBRATION from the Main Menu and then select LINE CALIBRATION to generate a display.

Below are some guidelines for determining whether the results are acceptable:

Do the %RSD's look acceptable for various concentrations?
Is the correlation coefficient larger than 0.995?

If the calibration results are acceptable, type A and press ENTER. A "New calibration coefficients stored" message will be displayed at the bottom of the screen and you can begin running samples.

9.11 Check Standards

This option allows you to verify that the calibration has not drifted. To check standard concentrations:

9.11.1 From the Main Menu, select CALIBRATION and then select CHECK STANDARDS. The check standard screen will appear.

9.11.2 Type 1 for a check standard blank. Enter, in units specified on the standards page, the range of acceptance.

9.11.3 Type 2 for check standards cup 2. Type the concentration and Enter. Type the percent acceptance and Enter.

9.11.4 Repeat this for up to seven check standards.

9.11.5 From Main Menu, select AUTOSAMPLES, then select SETUP and then check Enter the C1 frequency (e.g., 5/EPA protocol)

9.11.6 Halt: Enter Y if the instrument should halt after an unacceptable check standard. Enter N for an alert only. Macros can be written to automatically recalibrate and rerun samples if check standards fall outside specifications.

9.12 Running Samples

9.12.1 Press the F8 macro key. The Autosamples setup menu appears and a "Press F8 again to run sample" message will be displayed at the bottom of the screen.

9.12.2 Press the F8 A Macro key again. The PS200 will run the samples, print the results, and store the data in the folder you created.

NOTE: Each sample takes approximately 2 minutes to run: a full tray (88 samples) will take approximately 2 1/2 hours to complete. As operation is fully automatic, laboratory personnel need not be present while samples are running.

9.12.3 When all samples have been run, the system will beep and the word "Idle" will appear in the State field at the top of the screen. At this time, you can repeat steps detailed Sections 6.1-6.6 to run more samples or you can shut down the instrument. Refer to Section 9.13 for shutdown procedures.

9.13 Shutdown Procedures

There are two methods for shutting down the PS200. Under routine operation, when the system is used daily, only the lamp is shut off (system power remains on) and the Overnite routine is used to put the unit into a "sleep mode". If the system is to be completely turned off and not used for an extended period of time , or if it is to be shipped or moved, you must use the long-term Shutdown routine instead. These two methods are described below. For weekends or periods of "sleep" greater than 24 hours it is recommended to turn off the mercury lamp using the blue button.

NOTE: Before shutting down the instrument, the system must have beeped to indicate completion of the last procedure, and the word "Idle" should appear in the "State field in the top left of the displayed screen.

9.14 Short-Term (Overnite Macro)

Press the F2 macro key, type OVERNITE, and press ENTER. Turn off power to the lamp if the instrument will not be used for longer than 24 hours. In overnite mode, the pump and gas flow will turn on every few minutes, run for a few seconds and then stop. This cycle exercises the tubes so they don't get flat spots and fatigue, and the gas flow keeps the optical cell dry.

SUGGESTION: If you will be automating your run procedures with macros, call the Overnite procedure at the end (CM....) so that the system will shut down automatically when the last procedure is finished.

9.15 Long-Term (Shutdown Macro)

The Shutdown macro procedure is designed to flush out all lines with DI water to get rid of any chemical residues.

· Lift the sample tip and remove the rinse tray. Rinse and fill it with DI water and replace the tray. Lower the sample tip into the cleaned tray.

· Remove the reductant bottle cap and line and carefully place the tip of the line in the rinse tank (rest the cap on the corner of the tray).

· Turn off the lamp.

· Press the F2 macro button. Type SHUTDOWN and press ENTER. When you hear a beep and the word "Idle" appears in the State field at the top left of the screen (you will have to wait several minutes), release all pump clamps.

· Remove the front cover of the PS200 and remove the optical cell (refer to Section 6.0). Disconnect the two gas lines on the left side of the cell and leave them hanging. Replace the optical cell and the front cover.

NOTE: The next time you start up the system, you must remember to re-open the front cover, remove the optical cell and reconnect the gas lines.

· Shut off power to the computer, monitor, printer, and finally the PS200.

10.0 CALCULATIONS

Perform a linear regression or quadratic fit analysis of the calibration standard results. Compare sample results to the curve to determine the mercury concentration.

10.1 Water

ug/L Hg = ug/L x Dilution Factor

10.2 Soil

$$\text{mg/kg Hg} = \frac{\text{(ug/L) x L x Dilution Factor}}{\text{wt(g) x fraction solids}}$$

11.0 QUALITY CONTROL

11.1 Calibration curve must be composed of a minimum of a blank and five standards. A least square fit linear calibration curve must have a minimum correlation coefficient of 0.995, which must be reported with the raw data.

11.2 Calibration verification will be performed with a calibration blank and a continuing calibration verification (CCV) standard every ten samples and at the end of the analysis. The CCV must not vary more than 20% from its true value and must be prepared from a different source than the calibration curve standards.

11.3 Dilute samples if they are more concentrated than the highest standard or if they fall on the plateau of a calibration curve (dilute with a digested blank containing all reagents, or repeat the analysis using a smaller sample volume).

11.4 A minimum of one preparation blank must be analyzed per sample batch to determine if contamination has occurred. For this parameter, the continuing calibration blank (CCB) and preparation blank are equivalent.

11.5 Duplicate laboratory control samples (LCS) will be included with each sample batch of 20 samples. The analyzed result must not vary more than 20% from the true value. For this parameter, the LCS and CCV standard are equivalent.

11.6 Matrix spike and duplicate samples must be analyzed as requested by the client.

12.0 CORRECTIVE ACTIONS

When an out of control situation occurs, the analysts must use his/her best analytical judgment and available resources to determine the corrective action to be taken. The out of control situation may be caused by more than one variable. The analyst should seek the assistance of his/her immediate supervisor, QA personnel, or other experienced staff if he/she is uncertain of the cause of the out of control situation. The test must not be resumed until the source of the problem and an in-control status is attained. All samples associated with the out of control situation should be reanalyzed. Out of control data must never be released without approval of the supervisor, QA personnel or the laboratory manager.

Listed below are steps to be taken when an out of control situation occurs. The analyst must:

- demonstrate that all the problems creating the out of control situation were addressed
- document the problem and the action which was taken to correct the problem on a corrective action report form
- document on the corrective action report that an in control has been achieved and receive approval (signature) of the unit leader, QA personnel, or the laboratory manager prior to the release of any analytical data associated with the problem.

12.1 Suggested Actions to specific out of control situations

12.2.1 Calibration Curve

- reanalyze the standard curve;
- prepare new stock and/or working standards;
- check reagents/solutions and prepare fresh if necessary.

12.2.2 Initial Calibration Verification (ICV)

- repeat ICV to verify proper preparation;
- prepare new ICV from original stock;
- recalibrate with a new standard curve;
- prepare new stock and/or working standards;
- check reagents/solutions and prepare fresh if necessary.

12.2.3 Initial Calibration Blank (ICB)

- prepare new ICB to verify proper preparation;
- verify that the instrument base-line is stable and perform necessary maintenance, cleaning, etc.. to achieve stability;
- determine the source of contamination by the process of elimination, carryover from a previous analysis or reagent contamination and correct the problem;
- check reagents/solutions and prepare fresh if necessary;
- correct for any contamination and reanalyze ICB and any associated samples.

12.2.4 Laboratory Control Standards (LCS)

If LCS is low:
· reanalyze LCS to verify that it is out of control;
· determine the source of error within the preparation procedure, repeat the sample set, write a CAR.

If the LCS is high:
· reanalyze LCS to verify that it is out of control;
· determine the source of error within the preparation procedure, repeat the sample set;
· determine if the high result is due to contamination;
· check for contamination of reagents, LCS stock solution, or preparation area;
· correct for contamination, reanalyze.

12.2.5 Laboratory Control Standard Duplicate (LCSD)

Must meet all requirements and control limits as LCS in addition to limits set for precision

Precision: If precision is out of control, initiate the same actions specified for LCS

12.2.6 Preparation Blank (PB)

· reanalyze Prep Blank to verify that it is beyond the detection limit;
· determine the source of contamination;
· determine if the high result is due to contamination;
· check for contamination of reagents or preparation area;
· correct for contamination, reanalyze set;
· in the extreme case where all samples in the set are at least ten times greater than the PB, reanalysis will not be required. However, a corrective action report and approval will be necessary.

12.2.7 Matrix Duplicate (DUP)

- the sample must be reprocessed and reanalyzed;
- if the reanalysis results in data that is still out of the control limit, then the sample will be ticked with a "*";
- regardless of the outcome of the reanalysis, a CAR will be written and approved by the Unit Leader or Section Manager.

12.2.8 Matrix Spike (MS)

- the sample must be reprocessed and reanalyzed;
- if the reanalysis results in data that is still out of the control limit, then the sample will be ticked with a "N";
- regardless of the outcome of the reanalysis, a CAR will be written and approved by the Unit Leader.

12.2.9 Continuing Calibration Verification (CCV)

- repeat CCV to verify proper preparation;
- prepare new CCV from original stock;
- check for instrument base-line drift or a change in one or more of the reagents;
- check reagents/solutions and prepare fresh if necessary;
- recalibrate with a new standard curve and repeat all samples since the previous in control CCV;
- never dispose of any samples until you are sure that all QC, especially the CCV, are within the control limits.

12.2.10 Continuing Calibration Blank (CCB)

- prepare new CCB to verify proper preparation;
- verify that the instrument base-line is stable and/or perform necessary maintenance, cleaning, etc.. to achieve stability;
- determine the source of contamination by the process of elimination, carryover from a previous analysis or reagent contamination and correct the problem,
- check reagents/solutions and prepare fresh if necessary;
- correct for any contamination and reanalyze CCB and any associated samples;

· never dispose of any samples until you are sure that all QC, especially the CCB are within the control limits.

12.2 Summary

12.2.1 If any of the ICV, ICB, CCV or CCB results are out of control for any element, the instrument is restandardized and the samples associated with the out of control elements are reanalyzed.

12.2.2 If the PB or LCS are out of control for any element, the samples are redigested. An exception is if the sample concentrations are $\geq$ 10X the PB contamination, the results are reported as is.

12.2.3 If any of the Matrix Duplicate or Matrix Spike results are out of control, a reanalysis is performed if there is sufficient sample. If there is insufficient sample, or the reanalysis is still out of control, the client is notified of the poor results via a case narrative that is sent with the data report.

12.2.4 Corrective Action Report (CAR) forms are available for poor PB, LCS matrix dup, and matrix spike problems. These forms are completed by the analyst performing the analysis. The forms are then reviewed and signed by the unit leader. The signed forms are kept on file in the QC Dept.

13.0 HEALTH AND SAFETY

As always, general laboratory safety practices should always be followed. Waste samples should be handled with care due to the uncertainty of the properties and contents involved.

Fully fastened lab coat, safety glassed and latex gloves must be worn.

All chemical containers should be clean and properly labeled.

Immediately cleanup any materials spilled on the floor, in hoods or on bench tops.

All damaged or broken glassware should be discarded immediately.

Refer to the specific MSDS for the hazardous properties of any chemical or reagent involved in this procedure.

Acids should be handled with care.

The standard contains potentially harmful levels of mercury. Care should be taken to avoid contact with the stock solutions. Wash hands well if contacted.

INORGANIC ANALYSIS PROTOCOL
Mercury Analysis by Leeman AutoAnalyzer
EPA Methods 245.1; SW-846 Method 7470/7470; USEPA Document No. ILM02.1

These Approval Signatures Are Kept on File
with WESTON®'s Analytics Division
QA Standard Practice Records

REVISION NUMBER: 02

	Printed Name:	Signature/Date:
Written by:	Cheryl Boyd Metals Specialist	Cheryl L. Boyd 3/19/93
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Historical File: Revision 00: 10/03/90
Revision 01: 08/09/91
Revision 02: 03/19/93

Reasons for Change, Revision 02:

- Correction of water bath temperature monitoring at 90-95°C, rather than 95°C.
- Addition of SW-846 and USEPA CLP References.

INORGANIC ANALYSIS PROTOCOL
Nitrate-Nitrogen by Lachat QuikChem AE: Cadmium Reduction of Nitrate to Nitrite

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REVISION NUMBER: 00

1.0 PURPOSE

This method was used to determine the amount of nitrogen as nitrate-nitrite in a given sample. Nitrate-nitrogen alone can be determined by subtracting the nitrite-nitrogen amount determined by a separate procedure.

2.0 REFERENCES

This SOP was written using EPA 600/4-79-020 Method 353.2. Instrument specific instructions are taken from Lachat Method 10-107-04-01-C.

The Lachat QuikChem AE Auto Analyzer is used in place of the Technicon Auto Analyzer described in the method.

3.0 METHOD SUMMARY

A clear sample is passed through a column containing granulated copper-cadmium to reduce nitrate-nitrogen to nitrite-nitrogen. The nitrite-nitrogen (that originally was present plus reduced nitrate-nitrogen) is determined by diazotizing with sulfanilamide and coupling with N-(1-naphthyl)-ethylenediamine dihydrochloride to form a highly colored azo dye which is measured colorimetrically.

4.0 INTERFERENCES

4.1 Residual chlorine can oxidize the cadmium column and reduce its efficiency.

4.2 Low results may be obtained from samples containing high amounts of iron, copper or other metals. The addition of EDTA in the buffer helps reduce this interference.

4.3 Samples containing high amounts of organics, including oil and grease, will coat the cadmium granules. Pre-extraction with an organic solvent should be performed on these samples.

4.4 Turbidity should be removed by filtration through a 0.45 um pore diameter membrane prior to analysis.

5.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Sample containers, preservation techniques, and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client request. Samples should be preserved to a pH < 2. Listed below are the holding times and the references which include container and preservation requirements for compliance with the Clean Water Act (CWA) and the Safe Drinking Water Act (SDWA).

Regulation	Holding Time	Reference
CWA	28 days	40 CFR, Part 136.3
SDWA	28 days	EPA 600/4-79-020

6.0 INSTRUMENTATION AND EQUIPMENT

6.1 Lachat QuikChem AE System

Wavelength: 520 cm
Cell Path Length: 1.0 cm
Sample Loop Size: 17 cm
Cycle Period: 40 sec*
Inject to Start Period: 19 sec*

*These conditions can be adjusted to optimize instrument conditions.

6.2 Glassware/Miscellaneous

- disposable test tubes
- disposable 5 cc syringes
- 0.45 um filters (syringe or membrane)
- 100 mL volumetric flasks
- eppendorf pipets and tips

7.0 PREVENTATIVE MAINTENANCE

7.1 Change all pump tubing, manifold tubing, O-rings, and transmission tubing monthly.

7.2 Clean the surface of the instrument daily and wipe up all spills immediately.

7.3 Download the files to a disk on a monthly basis.

7.4 Check diagnostic when instrument is turned on for valve function, sample function, and detector function. Response to problems immediately.

7.5 Consult Lachat manuals for instructions on how to diagnose problems.

8.0 STANDARD AND REAGENTS

All standards and reagents are prepared with Type II Deionized Water, unless otherwise stated, in Class A volumetric flasks.

8.1 Sulfanilamide Color Reagent

To 600 mL DI water in a 1.0 L volumetric flask, add 100 mL 85% phosphoric acid, 40 g sulfanilamide, and 1 g N-(1-naphthyl)ethylenediamine dihydrochloride. Dissolve the mixture by stirring on a stir plate for 30 minutes. Dilute to volume and mix again.

- Life of Reagent: one month
- Storage Requirements: store in the dark

8.2 Ammonium Chloride Buffer

To 800 mL DI water contained in a 1.0 L volumetric flask, dissolve 85 g ammonium chloride (NH_4Cl) and 1.0 g disodium ethylenediamine tetracetic acid dihydrate ($Na_2EDTA \cdot 2H_2O$). Adjust pH to 8.5 with 15 N sodium hydroxide and dilute to volume.

- Life of Reagent: one year
- Storage Requirements: none

8.3 15 N Sodium Hydroxide

Slowly dissolve 150 g sodium hydroxide in 250 mL DI water. Cool.

- Life of Reagent: one year
- Storage Requirements: Store in a plastic bottle.

8.4 2% Copper Sulfate

In a 1.0 L volumetric flask, add 20 g copper sulfate ($CuSO_4 \cdot 5H_2O$) to ~800 mL DI water. Mix and then dilute to volume with DI water.

- Life of Reagent: one year
- Storage Requirments: none

8.5 Acetone

Purchased from a chemical vendor.

- Life of Reagent: manufacturers recommendation
- Storage Requirements: store in a flammables cabinet

8.6 1 M Hydrochloric Acid

To a 100 mL volumetric flask containing approximately 80 mL DI water, add 8 mL hydrochloric acid (HCl). Mix and dilute to volume with DI water.

- Life of Reagent: one year
- Storage Requirment: none

8.7 Nitrate Stock Solution I, 100 mg N/L

To a 1.0 L volumetric flask containing approximately 800 mL DI water, add 0.722 g potassium nitrate (KNO_3) and 2 mL chloroform ($CHCl_3$). Mix and dilute to volume with DI water.

- Life of Reagent: six months
- Storage Requirements: keep refrigerated

8.8 Nitrate Stock Solution II, 100 mg N/L

Prepare as Reagent 8.7 using an alternate source of potassium nitrate.

- Life of Reagent: six months
- Storage Requirements: keep refrigerated

8.9 Nitrite Stock Solution III, 100 mg N/L

To a 1.0 L volumetric flask containing approximately 800 mL DI water, dissolve 0.4926 g potassium nitrite (KNO_2). Preserve with 2 mL chloroform ($CHCl_3$). Mix and dilute to volume with DI water.

- Life of Reagent: six months
- Storage Requirements: keep refrigerated

8.10 Cadmium Granules, 0.3 - 15 Mesh

Purchased from a chemical vendor. CAUTION: Extremely Toxic.

- Life of Reagent: manufacturers recommendation
- Storage Requirements: none

9.0 PROCEDURE

9.1 Reporting Limit

Waters..... 0.1 mg N/L
Wastes..... 1 mg N/kg

9.2 Sample Size

Waste samples:	10 g/100 mLs DI water
Sludge & Oil Samples:	*10 g/100 mLs DI water
Water samples:	10 mLs

*More sample may be used for sludges high in water content when a low dry weight reporting limit is required.

9.3 Cadmium Column Preparation

9.3.1 Weigh 10 g of cadmium granules into a 250 mL beaker. WEAR GLOVES since cadmium is extremely toxic.

9.3.2 Wash the cadmium with several 50 mL aliquots of DI water. Save the washings for proper disposal!

9.3.3 Wash the cadmium with a 50 mL portion of acetone followed by two DI washings. Save the washings for proper disposal!

9.3.4 Wash the cadmium twice with 50 mL portions of 1 M hydrochloric acid (HCl). The cadmium should have a silver sheen. Rinse it several times with DI water. Save all the rinses for proper disposal!

9.3.5 Add 100 mL 20% copper sulfate to the cadmium and swirl. Allow to sit at least 5 minutes. If the copper sulfate has lost its blue color, repeat the rinse with fresh copper sulfate. If not and the cadmium appears to be dark grey/black, rinse several times with ammonium chloride buffer (Rgt. 8.2). Store the cadmium covered with ammonia chloride buffer.

9.4 Packing the Cadmium Column

WEAR GLOVES!

9.4.1 Unpack any cadmium currently present in the column. Reserve the foam ends for further use and properly dispose of the used cadmium.

9.4.2 Set up green pump tubing with transmission tubing, tubing converter, a union fitting, and 0.032 ID tubing. Place the assembled tubing into the pump.

Place the transmission tubing into the container of ammonia chloride buffer. Turn the pump on and fill the tubing with buffer. Turn the pump off. Attach the empty cadmium column to the buret stand. Place one foam plug into the bottom of the empty column. Attach one end fitting union connector and a small piece of 0.032 ID tubing to the column. Plug the small amount of tubing into the union connector which is attached to the reagent line. Turn the pump back on and fill the whole system. Turn the pump off. Check to see that the meniscus of the ammonium chloride rises above the column.

9.4.3 Using a small scoop, carefully add treated cadmium to the empty column. Tap the column with the handle of a screw driver to pack the cadmium tightly. When the level of cadmium reaches about 5 mm from the top, insert the second foam plug and attach the other end fitting. Turn on the pump and fill the added tubing. Check the flow through the column by setting the speed of the pump to 35 and collecting the fluid coming from the column. The rate should be greater than 4 mL/min. Shut the pump off and carefully attach the end fitting together at the union. Take care not to entrap air into the system. The filled cadmium column should weigh 36 - 36.5 g.

9.5 Manifold Preparation

Place the manifold on the proper Lachat channels. Attach all the tubing as shown in the manifold diagram. Turn the main instrument and the pump on. Check the main instrument display for proper functioning. Turn on the Master Computer and download the proper method. Insert the transmission tubing into the respective solutions. When all tubing is filled and you are certain there are no leaks, attach the prepared cadmium column. Great care needs to be taken so that air does not enter the column. Follow these directions:

9.5.1 Make sure all reagents are pumped into the manifold and no air is present.

9.5.2 Turn the pump off.

9.5.3 On the column, disconnect the center tubing from one of the union connectors and immediately connect it to the outlet tubing of the buffer mixing coil.

9.5.4 Connect the open tubing on the column to the tee fitting where the coloring reagent is added.

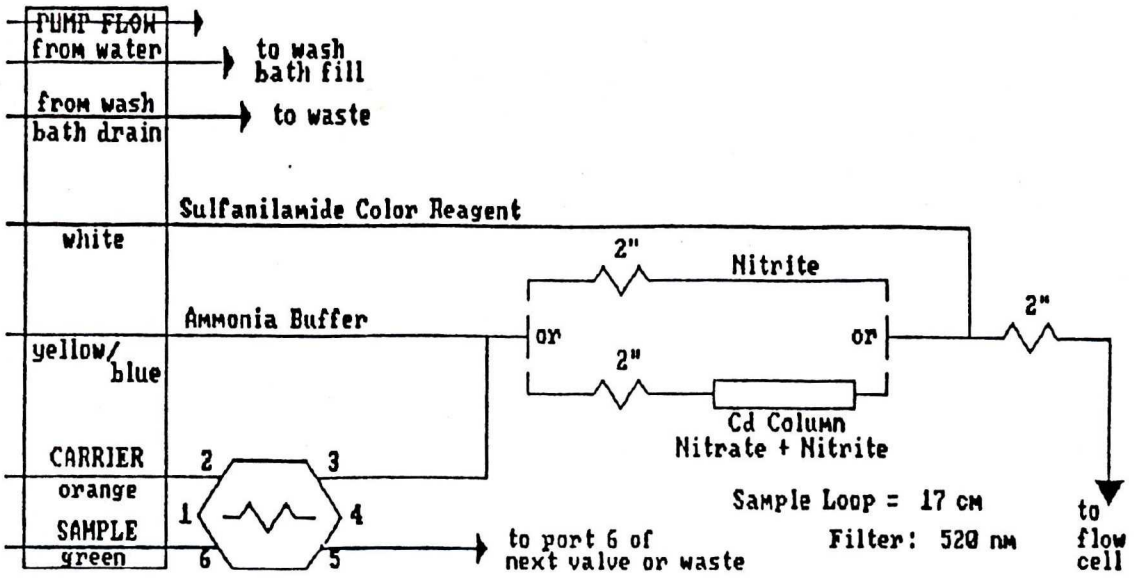
9.5.5 Turn the pump on at normal speed.

9.5.6 Direction of flow through the column is irrelevant.

If air enters the column, it can lower the efficiency of the column. Small bubbles may not interfere, but if reduction is not adequate, this is quite often the problem.

Manifold Diagram

Manifold Diagram:



CARRIER is water.

2" is **135** cm of tubing on a 2 in coil support

APPARATUS: Standard valve, flow cell, and detector head modules are used.

All manifold tubing is **0.8 mm (0.032 in)** i.d. This is 5.2 uL/cm.

MANIFOLD DIAGRAM REVISION DATE: 30 December 1986

Reference: QuikChem Method 10-107-04-1-C

9.6 Calibration Procedure

Instrument calibration consists of two types: Initial Calibration and Continuing Calibration.

9.6.1 Initial Calibration

Establishes the calibration range of the instrument and determines the instrument response over that range.

The autoanalyzer will be calibrated prior to each day of use. The calibration standards will be prepared from reference materials appropriate to the analyses being performed, and working standards will include five (5) concentraations which cover the anticipated range of measurement. One of the calibration standards will be at the desired detection limit. Additionally, a calibration blank will be analyzed. The requirement for an acceptable initial calibration will be a correlation coefficient equal to or greater than 0.995 in order to consider the response linear over the measured range.

If the correlation coefficient criteria of 0.995 is not met, the instrument will be recalibrated prior to analysis of samples. Calibration data, to include the correlation coefficient, will be entered into the laboratory notebook with the sample data to maintain a permanent record of instrument calibrations.

Before sample analysis, an Initial Calibration Verification (ICV) Standard is analyzed. The response calculated as percent recovery of this standard must be within +/- 10% of the true vaue or the instrument is recalibrated. The response of the Initial Calibration Blank (ICB) must be less than the reporting limit.

9.6.2 Continuing Calibration

Used within an analytical sequence to verify stable calibration throughout the sequence, and/or to demonstrate that instrument response did not drift during a period of non-use of instrument.

A Continuing Calibration Verification (CCV) and Blank (CCB) will be analyzed at a frequency of 10% and at the end of the analysis sequence. The response, calculated as a percent recovery of the true value, must be +/- 10%

of the true value. The response of the CCB must be less than the detection limit.

9.7 Analytical Sequence

Calibration Controls	Sequence	Control Limit
Standards	prior to samples	+/-10%
Corr. Coeff.	prior to samples	$\geq$ 0.995
Column Efficiency Std.	prior to samples/prior to ICV/ICB	90 - 110%
Init. Cal. Ver. (ICV)	prior to samples/after calibration	90 - 110%
Init. Cal. Blk. (ICB)	prior to samples/after calibration	< Reporting Limit
Cont. Cal. Ver. (CCV)	every 10 readings	90 - 110%
Cont. Cal. Blk. (CCB)	every 10 readings	< Reporting Limit

Quality Controls	*Frequency	Control Limit
Prep. Blk. (PB)	1 in 20 samples	< Reporting Limit
Lab. Control Std. (LCS)	1 in 20 samples	80 - 120%
LCS Duplicate (LCSD)	1 in 20 samples	80 - 120%
Matrix Duplicate	1 in 20 samples	$\leq$ 20% RPD
Matrix Spike	1 in 20 samples	75 - 125%

*Drinking Water samples are analyzed in sets of 10 with a duplicate and spike performed on the drinking water matrix. Control limits are $\leq$ 20% RPD for duplicates and +/- 15% for spikes.

9.8 Standards Preparation

(Use volumetric flasks to prepare the following standards.)

Daily Standard ID	Concentration (ppm)	Volume of Standard I (Rgt. 8.7)
A	2.0	2 mLs diluted to 100 mLs
B	1.5	1.5 mLs "
C	1.0	1 mLs "
D	0.5	0.5 mLs "
E	0.2	0.2 mLs "
F	0.1	0.1 mLs "
G	0.0	0.0 mLs "

9.9 Quality Control Standards

Quality Control	Volume of Standard II (Rgt. 8.8)	Concentration
Lab. Control Std. (LCS)	1.0 mLs / 100 mLs DI Water	1.0 ppm
LCS Duplicate	1.0 mLs / 100 mLs DI Water	1.0 ppm
ICV/CCV	1.0 mLs / 100 mLs DI Water	1.0 ppm
Matrix Spike	1.0 mLs / 100 mLs DI Water	1.0 ppm

9.9.1 1.0 ppm Nitrite Standard for Column Efficiency

Dilute 1.0 mL of Nitrite Stock Solution III (Rgt. 8.9) to volume with DI water in a 100 mL volumetric flask. Analyze in sequence (See Section 9.7.) The recovery acceptance limit is 90 - 110%. When the column efficiency standard recovery is calculated against the ICV concentration, the acceptance limit of 90 - 110% must be met as well.

9.10 Sample Preparation and Analysis

9.10.1 Water Samples

9.10.1.1 Pour approximately 10 mL of sample into a test tube. Adjust the pH to neutral with 15 N sodium hydroxide and place in the rack.

9.10.1.2 If turbidity is present, filter the sample through a 0.45 um membrane filter prior to analysis.

9.10.1.3 If oil & grease is present, extract the sample with freon prior to analysis.

9.10.2 Waste/Soil Samples

9.10.2.1 Weigh 10 g of sample, to the nearest 1.0 g, into a 250 mL beaker. Add 100 mL of DI waters and stir on a stir plate for approximately 30 minutes.

9.10.2.2 Filter the sample through a glass fiber filter, then through a 0.45 um membrane filter.

9.10.2.3 If oil & grease is present, extract the filtrate with freon prior to analysis.

9.10.3 Colorimetric Analysis

9.10.3.1 Double check the order of samples on the rack with the tray identification.

9.10.3.2 Submit the tray for analysis.

10.0 CALCULATIONS

Terminology:

N_3N_2 Total nitrogen in the form of nitrate-nitrogen and nitrite-nitrogen obtained directly from the auto analyzer.

NO_3 Nitrogen in the nitrate-nitrogen form.

NO_2 Nitrogen in the nitrite-nitrogen form obtained from EPA Methods 354.1 or 300.0.

10.1 Nitrate

N_3N_2 mg/L = N_3N_2 (mg N/L curve) x dilution factor

NO_3 mg/L = N_3N_2 (mg N/L) - NO_2 (mg/L)

$$N_3N_2 \text{ mg/kg} = \frac{N_3N_2 \text{ (mg N/L curve)}}{\text{sample wt. (g)}} \times \text{final volume (mLs)} \times \text{dilution factor}$$

$$N_3N_2 \text{ mg/kg (dry weight)} = \frac{N_3N_2 \text{ (mg/kg wet wt.)}}{\% \text{ solids (as decimal)}}$$

10.2 Column Efficiency

$$\frac{1.0 \text{ mg N/L } N_3N_2 \text{ observed}}{1.0 \text{ mg N/L } NO_2 \text{ observed}} \times 100$$

10.3 Matrix Spike Recovery

$$\frac{\text{(matrix spike result - sample result)}}{\text{known concentration}} \times 100$$

10.4 % Relative Percent Difference

$$\frac{|\text{sample result - duplicate result}|}{\text{(sample result + duplicate result/2)}} \times 100$$

10.5 Reporting Results

Without rounding, enter the raw data on the appropriate LOTUS Spreadsheet. Carefully print, review and approve the spreadsheet before creating a print file and transferring the data to LIMS.

11.0 QUALITY CONTROL

11.1 One method blank and two Lab Control Standards (LCS) will be included in each laboratory lot of 20 samples. Regardless of the matrix being processed, the LCS and method blanks will be in an aqueous media.

11.2 The method blank will be examined to determine if contamination is being introduced in the laboratory.

11.3 The LCS's will be examined to determine both precision and accuracy.

11.4 Accuracy will be measured by the percent recovery (%R) of the LCS. The recovery must be in range, as determined by in-house control limits or statistical analysis, in order to be considered acceptable. Additionally, %R will be plotted on control charts to monitor method accuracy.

11.5 Precision will be measured by the reproducibility of both LCS's and will be calculated as relative percent difference (%RPD). Results must agree within in-house control limits or statistical control limits in order to be considered acceptable.

11.6 One matrix spike and matrix duplicate is performed per matrix per 20 sample analytical set. Results must agree within the in-house precision/accuracy limits or statistical control limits in order to be considered acceptable.

12.0 CORRECTIVE ACTIONS

When an out of control situation occurs, the analysts must use their best analytical judgment and available resources to determine the corrective action to be taken. The out of control situation may be caused by more than one variable. The analyst should seek the assistance of their immediate supervisor, QA personnel, or other experienced staff if they are uncertain of the cause of the out of control situation. The test must not be resumed until the source of the problem and an in-control status is attained. All samples associated with the out of control situation should be reanalyzed. Out of control data must never be released without approval of the supervisor, QA personnel or the lab manager.

12.1 Listed below are steps to be taken when an out of control situation occurs. The analyst must:

- demonstrate that all the problems creating the out of control situation were addressed;
- document the problem and the action which was taken to correct the problem on a corrective action report form;
- document on the corrective action report that an in control has been achieved; and
- receive approval (signature) of the Section Manager, Unit Leader, QA personnel, or the Laboratory Manager prior to the release of any analytical data associated with the problem.

12.2 Suggested Actions to specific out of control situations:

12.2.1 Calibration Curve

- reanalyze the standard curve;
- prepare new stock and/or working standards;
- check reagents/solutions and prepare fresh if necessary.

12.2.2 Initial Calibration Verification (ICV)

- repeat ICV to verify proper preparation;
- prepare new ICV from original stock;
- check for instrument base-line drift;
- restandardize with existing standards, reanalyze;
- check reagents/solutions and prepare fresh if necessary;
- prepare new stock and/or working standards and recalibrate;

12.2.3 Initial Calibration Blank (ICB)

- prepare a new ICB to verify proper preparation;
- verify that the instrument base-line is stable and/or perform necessary maintenance, cleaning, etc... to achieve stability;
- determine the source of contamination by the process of elimination, correct the problem and reanalyze. (Carry over from a previous analysis or reagent contamination are two common sources).

12.2.4 Laboratory Control Standards (LCS)

If either LCS1 or LCS2 exceeds acceptance limits:
- reanalyze LCS to verify that an out of control situation exists;
- determine the source of error within the preparation procedure, correct the problem and repeat the sample set. (Sources of contamination could be either the reagents, the LCS stock solution, or the preparation area.)

Precision: LCS1 and LCS2 must meet the control limits of $\leq$ 20% RPD. If this criteria is not met, and both LCS's meet the % Recovery control limits, then see your Section Manager or Unit Leader for proper corrective action.

12.2.5 Preparation Blank (PB)

- reanalyze PB to verify contamination at a level > Reporting Limit;
- determine the source of contamination and correct the problem;
- all samples whose concentration is <10 times the PB level must be reprocessed and reanalyzed; any sample which is >10 times the PB level need not be reanalyzed. However, a corrective action report must be filled out and approval obtained.

12.2.6 Matrix Duplicate (DUP)

- the sample must be reprocessed and reanalyzed unless the sample concentration is <5 times the Reporting Limit, then the ± Reporting Limit rule applies;
- if the reanalysis is within the control limits, the second value is reported;
- if the reanalysis is still outside of the control limits, a CAR must be written and then approved by your Section Manager or Unit Leader.

12.2.7 Matrix Spike (MS)

- the sample must then be reprocessed and reanalyzed unless the sample concentration exceeds the spike concentration by a factor of 4 times;
- the original spike results must be entered onto the spreadsheet with the "S" code even though the control limits were exceeded;
- the reanalysis result must be entered onto the spreadsheet using the "T" code regardless of whether it is within the control limits. There is no need to write a corrective action if both the "S" and "T" codes were entered into LIMS.

12.2.8 Continuing Calibration Verification (CCV)

- repeat CCV to verify proper preparation;
- prepare new CCV from original stock;
- check for instrument base-line drift;
- check reagents/solutions and prepare fresh if necessary;
- recalibrate with a new standard curve and repeat all samples since the previous in control CCV;
- never dispose of any samples until you are sure that all QC are within their designated control limits.

12.2.9 Continuing Calibration Blank (CCB)

- prepare a new CCB to verify proper preparation;
- verify that the instrument base-line is stable and/or perform necessary maintenance, cleaning, etc... to achieve stability;
- determine the source of contamination by the process of elimination, correct the problem and reanalyze all the samples since the previous in control CCB. (Carry over from a previous analysis or reagent contamination

are two common sources).
· never dispose of any samples until you are sure that all QC are within their designated control limits.

13.0 HEALTH AND SAFETY

As always, general laboratory safety practices should always be followed. Waste samples should be handled with care due to the uncertainty of the properties and contents involved. Refer to the specific MSDS for the hazardous properties of any chemical or reagent involved in this procedure.

Proper disposal procedures for cadmium need to be followed due its extreme toxicity.

Eff. Date: 01/15/93 Initiated By: QC Department Approved By: D. L. Harper Authorized By: A. M. Henry SP No. 21-15G-353.2

INORGANIC ANALYSIS PROTOCOL
Nitrate-Nitrogen by Lachat QuikChem AE: Cadmium Reduction of Nitrate to Nitrite

These Approval Signatures Are Kept on File with WESTON®'s Analytics Division QA Standard Practice Records

REVISION NUMBER: 00

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Historical File: Revision 00: 01/15/93

Reasons for Change, Revision 00:

- formalize the procedures used to determine Nitrate-Nitrogen by the Lachat.